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Chemical Engineering Progress

Volume 46
Number 11
NOVEMBER 1950

Nonmember chemical engineers who wish to know more about our Columbus meeting can obtain a complimentary copy of the program from the Executive Secretary's office, 120 East 41st Street, New York 17, N. Y.

Another Future Meetings listing for ready information begins this month in C.E.P. on local sections—name of section, meeting place and date, speaker and title of paper. (See p. 28.)

“... Engineers must be practical men, yet they must have the imagination to create new methods and the skills to use facts and logic in solving scientific problems. Research helps colleges assure these abilities...” No cliché, but rather the role of research in engineering education according to representatives of 83 leading engineering schools and defined in “Research is Learning,” a new nontechnical publication of the Engineering College Research Council of the A.S.E.E. The purpose in printing this 32-page pamphlet is to disseminate the idea more broadly that university research activities are directly related to educational programs and are in no way competing with profit-making research. Available without cost on request to the Secretary of the Engineering College Research Council, Room 7-204, 77 Massachusetts Avenue, Cambridge 39, Mass.

Chemical Engineering Progress is now available on microfilm and for libraries pressed for shelf space the complete current volume of Chemical Engineering Progress will be available after the year's end from University Microfilms. Under this plan the library must be a subscriber to C.E.P. in order to obtain the complete microfilm volume. In practice it is expected that the library will keep the printed issues unbound for two or three years. When the period of maximum use is over, the microfilm reel is substituted. The cost will approximate that of conventional binding. Inquiries should be sent to University Microfilms, 313 N. First Street, Ann Arbor, Mich.

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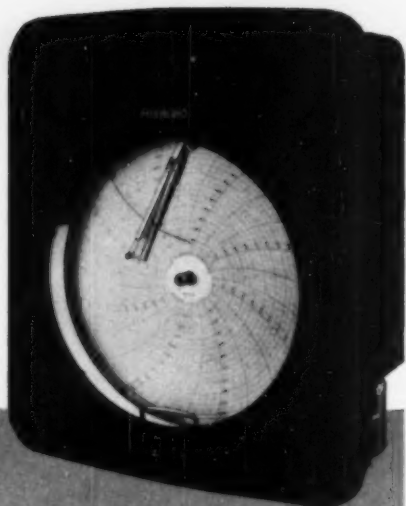
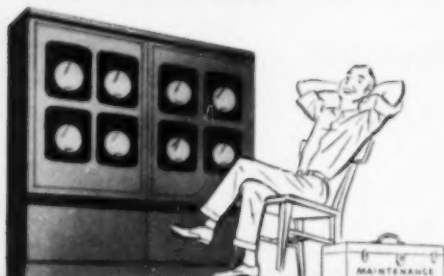
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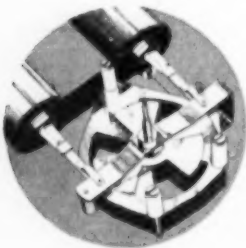
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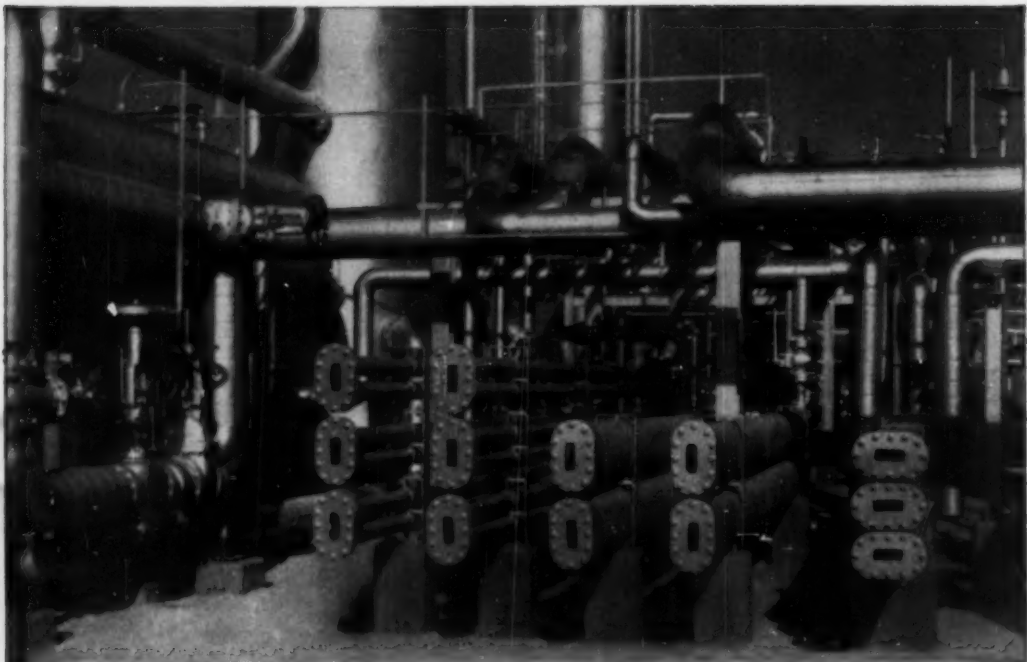
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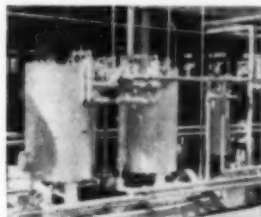
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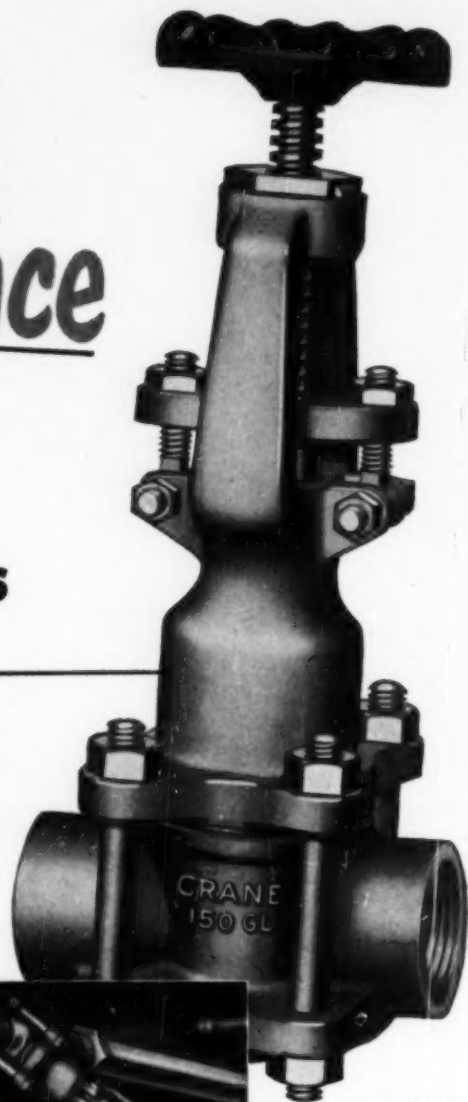
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(Right) Photo of subject installation

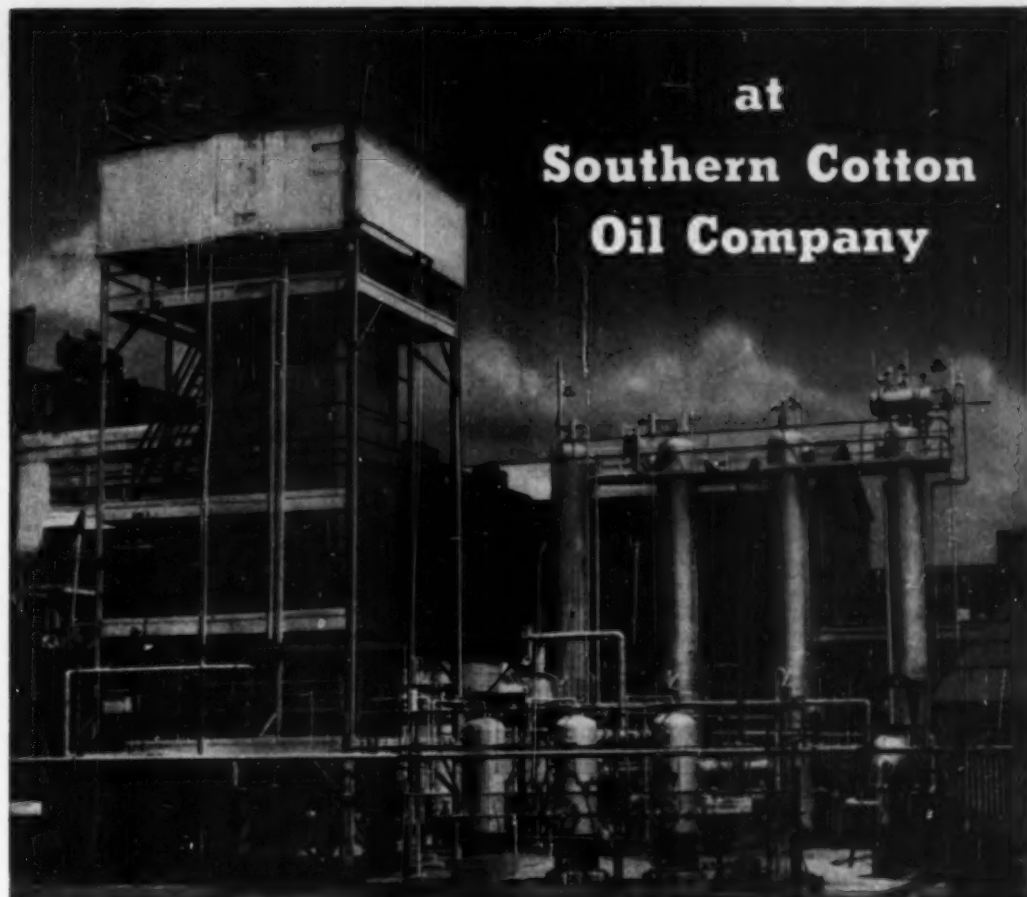


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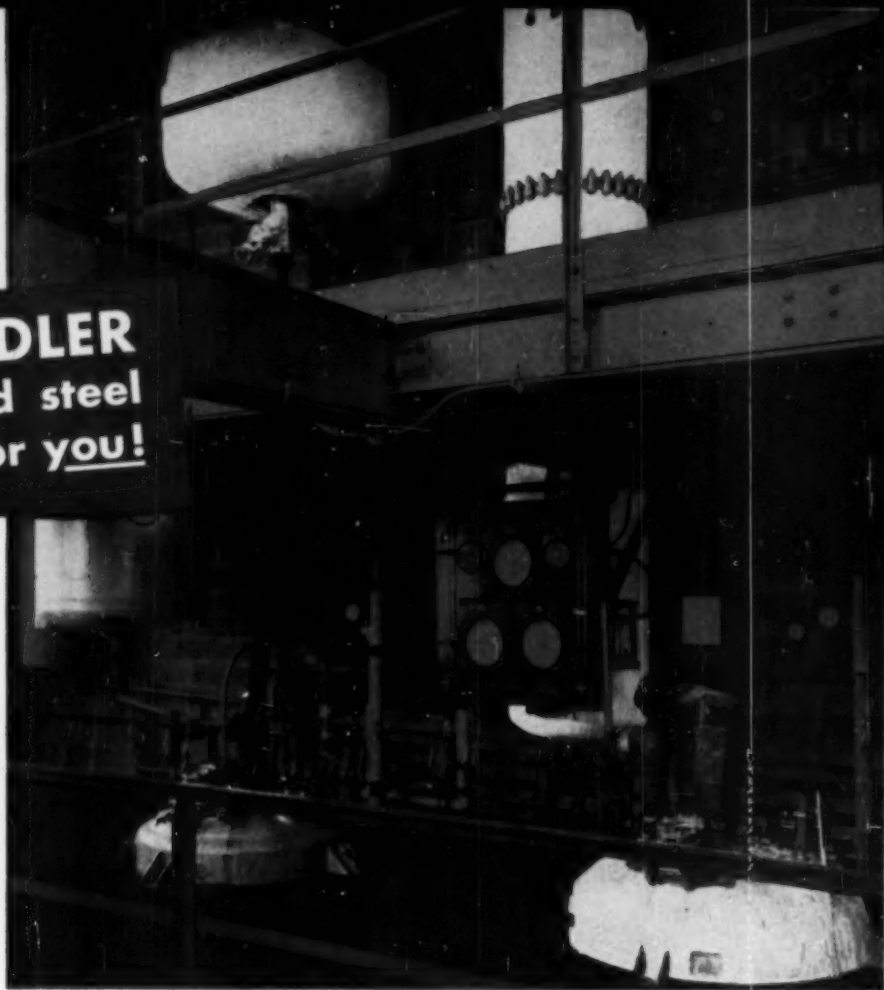
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OPINION AND COMMENT

THE SUPPLY OF ENGINEERS

DURING the past few years, engineering colleges have graduated a larger number of men with engineering training than in any other equivalent period. In 1948, 27,460 bachelors' degrees in engineering were granted by U. S. colleges, in 1949—45,200, and in 1950, 50,000. The number graduated in chemical engineering would be 3,984 in 1948, 4,700 in 1949 and 4,600 in 1950.

However, the demands of industry, owing to technological advances and generally improved business conditions have brought about a shortage of men with engineering training.

Considering the appreciable requirements for personnel by the armed forces this situation becomes more aggravated. Further, considering the fact that the age groups through which the Selective Service System operates is at present the same as that of college students and young graduates, the shortage could become acute since those presently employed, as well as those in training, might be inducted.

For a considerable period engineering college administrators have viewed with apprehension the steadily reducing freshman enrollments. If enrollments in the engineering colleges continue on this downward trend it is quite evident that in a few years the demand for young men with engineering training will greatly exceed the supply. Dean S. C. Hollister of Cornell University has just reported, at the annual meeting of Engineers' Council for Professional Development at Cleveland, Ohio, on Oct. 20, the results of a survey made this fall, and the figures which he has been able to compile are extremely interesting and point to the seriousness of the situation.

As a basis for his report the freshman engineering registration in 34 institutions was reviewed accurately and a study of previous enrollments showed that these 34 institutions represented 50% of the engineering enrollment in the country. Therefore it was logical to use this factor in estimating the present 1950 freshman enrollment.

As of Oct. 1, the survey indicated there were 26,500 freshmen registered in engineering curricula. This figure is in contrast to 36,508 registered at the same date in 1949, 47,672 in 1948, and 57,507 in 1947. It is interesting at this point to note that in the fall of 1940, there were 33,175 freshmen enrollments in engineering.

Another significant set of figures which Dean Hollister presented showed that in 1940, 2.7% of the high school graduates entered engineering; in 1947, 5.3%;

in 1948, 4%; in 1949, 2.9% and in 1950 only 2.2%. It is worthy of further note that because of the declining birth rate during the 1930's, high school enrollments are expected to decrease about 10% based on present figures, until the year 1953. It is expected that then there will be an uptrend, and a return to present figures should be reached about 1958.

To emphasize these facts Dean Hollister pointed out, that in spite of the large number of men graduating in June, 1950 (50,000), there is at the moment a shortage of trained engineering personnel. He estimated that in 1951 there will be only 32,500 graduates, in 1952, 21,900, in 1953, 17,000, and in 1954, 12,400. It was therefore extremely conservative of him to estimate that the demands of industry in peacetime call for at least 20,000 engineering graduates. He went on to estimate that the demands in the future, considering both civilian and military requirements, might well run as high as 30,000. If all these statistics are true, and I believe they are, it is cold substantiating data of absolutely no comfort, that we are facing a serious shortage of young men with engineering training.

From annual surveys made by the American Society for Engineering Education it can be estimated that the number of young men graduating with degrees in chemical engineering was about 4,600 in 1950. In 1951 it could be estimated that the graduating group will be about 3,800, in 1952 about 3,100. From the freshman enrollment, as of Oct. 1, 1949, the graduating group in 1953 will probably not exceed 2,000.

It therefore becomes the immediate duty of industry to review carefully the manner in which it is using its engineering personnel, and to make sure that these men are being employed at their highest skills. Companies using engineers should see to it that these men are trained for high responsibilities at as early a date as possible. If the demands on industry should increase because of an intensification of the present emergency, these men will have to take on more and greater responsibilities. It is quite evident from the above figures that replacements will not be available from the young men now in college. Furthermore every effort should be made to upgrade men with contributory skills, even though their training has not been as complete as that of the graduate engineer. And just as in the last military expansion, consideration should also be given to the training of women for the positions in the engineering field which they are qualified to fill.

S. L. Tyler, Secretary A.I.Ch.E.



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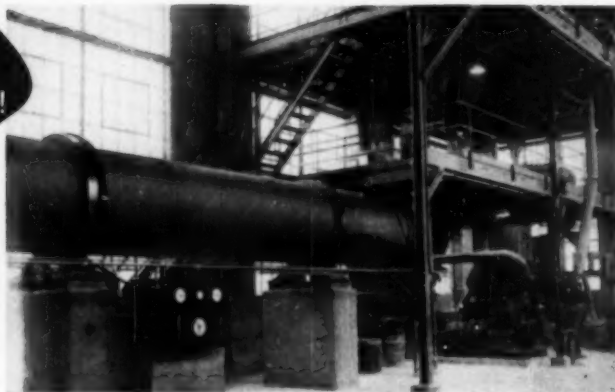
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The Sharples Corporation, Philadelphia, Pennsylvania

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Figure 1 illustrates the action of gravitational force on a three-phase system. In Figure 2, the same system is shown subjected to centrifugal force. Centrifugal force is able to perform similar types of operations that can be performed in a gravitational field, and for commercial purposes, a centrifuge may be defined as a device to generate a centrifugal field which can be used to (1) separate immiscible fluids, such as oil and water; (2) remove and recover

solids dispersed in fluids; (3) remove excess liquids from solids, and (4) any combination of the first three.

Types of Centrifuges

The basic types of centrifuges are:

1. Centrifugal "filter," in which the solid phase is supported on a porous surface, such as a screen, that permits passage of the fluid phase under the acceleration of centrifugal force.

2. "Settling machine," the gravitational equivalent of which is a settling tank, or thickener, which is often of highly modified form for specific service conditions.

Centrifugal fields are produced in commercial centrifuges that are many thousand times greater than the force of gravity. The centrifuge in general treats materials in less time than is required for the same degree of treatment under gravity, and in many cases produces quantitative results that cannot be attained in the gravitational field regardless of the time factor, because of the opposing forces of surface tension and diffusion.

Fairly complete mathematical analyses (1-3) have been made of the various types of settling-machine centrifuges. These are developed primarily from Stokes' law. The performance characteristics of centrifuges with widely different design features can be compared by the relationship of their sigma constants (2) and which is defined as the area of a gravity settling tank that is equivalent in sedimenting performance to the centrifuge under consideration.

For small particles such as protein and virus molecules, Svedberg's sedimentation constant (3), the specific sedimentation velocity referred to a unit or gravitational field and to water as a suspending medium and expressed in units of 10^{-13} /sec. has been found to be a useful value and again serves to tie centrifugal force to its gravitational analogue.

The mathematical analysis of the centrifugal filter is not nearly so complete. The formula $Y = e^{A+Bx}$, in which

Y = % of retained mother liquor

x = reciprocal of the time factor,

t , and A and B are constants for a given machine, appears to be generally applicable (4). No satisfactory analyses have been reported of the A and B factors, although it is known that both are affected by centrifugal force and crystal size. Other controlling factors are believed to be the thickness of the crystal bed, viscosity of the mother liquor, and the interfacial tension between mother liquor and crystals.

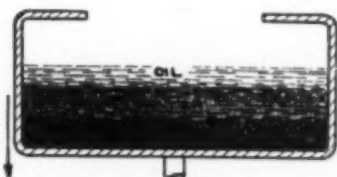


Fig. 1. Effect of gravity on a three-phase system.

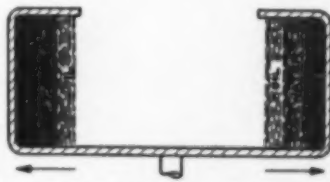


Fig. 2. Effect of centrifugal force on same system.

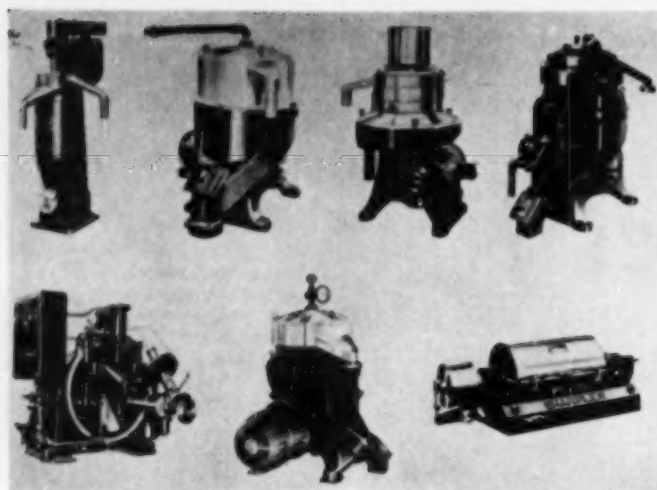


Fig. 3. Sharples centrifuges.

It will be seen from the foregoing that centrifuges of either category are operable on discontinuous systems and that for mathematical analysis, accurate measurements of the components are required.

In monodisperse systems, when such measurements are available or can be made, accurate forecasts of centrifuge performance are possible. This is clearly shown by the work of Svedburg and others. Such measurements are usually difficult or impossible to make in the

heterogeneous systems encountered in industry, and for even reasonable accuracy, centrifuge performance estimates have to be based on empirical pilot plant or actual full-scale tests.

In the chlorine-alkali industry, and in the utilization of its products, there are many centrifuge applications. Certain of these have been selected to exemplify the utility of centrifuges in the process industries. Figure 3 shows a few of the types of centrifuges available for commercial service.

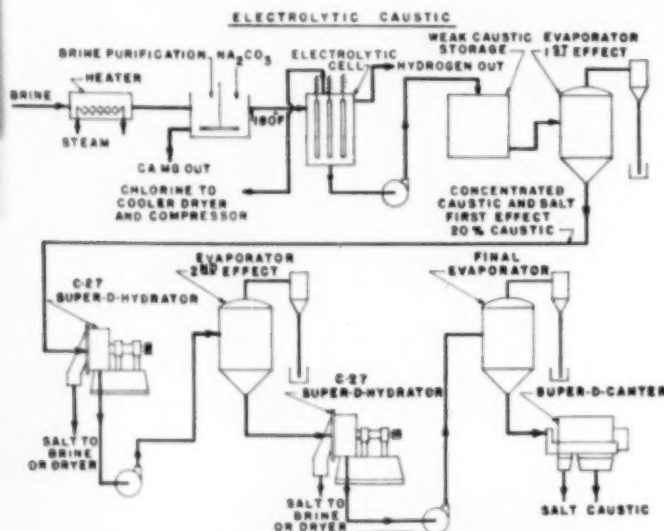


Fig. 4. Diagram of diaphragm cell plant (electrolytic chlorine-alkali production)

Figure 4 illustrates in simplified form, a diaphragm cell electrolytic caustic soda plant, showing the stages at which two different types of centrifuges are applied. Depending on the cell efficiency, the per cent conversion of salt to caustic and chlorine, between 1.0 and 1.5 tons of crystallized sodium chloride must be handled per ton of caustic soda produced. The salt removed from the caustic may be reused for making cell brine, or, in some instances, the salt may be dried for other use. In the latter case, the residual moisture content of the salt leaving the centrifuge is important. In either case, it is important to remove the salt at as low a residual caustic soda content as possible to minimize loss and rehandling. Reduction of the caustic content of the salt is accomplished in a centrifuge of the centrifugal filter type by rinsing the salt crystals with water to displace their adhering film of mother liquor.

The efficiency of the utilization of this rinse water is an important concept. All the rinse water used constitutes additional evaporator load, so there is a direct relationship between rinse water consumption and plant operating costs. Any rinse water beyond that immediately required to displace the adhering mother liquor from the salt will dissolve a corresponding amount of salt which then has to be rehandled, after evaporation, another added operating cost.

Figure 5 shows the cross section and Figure 6 an exploded view of an automatic batch centrifuge suitable for performing this operation. An installation of four of these centrifuges in an electrolytic caustic soda plant is shown in Figure 7. In this type of centrifuge, the perforate basket lined with wire screen is rotated on a horizontal axis continuously and at full speed, and the unit operations of loading the basket with salt, rinsing the salt cake, spinning to dryness and unloading are under time-cycle control of appropriate air diaphragm valves.

Centrifuges of this type are designed with a built-in cake distributor (part *H* of Fig. 5) that has a triple function:

It serves to distribute the cake from the entering feed slurry around the basket to keep the rotating element in balance.

It automatically closes the feed valve when the basket is filled with salt to prevent overloading regardless of the time-cycle setting.

It serves to distribute the cake uniformly across the face of the basket. This insures a crystal bed of uniform thickness which minimizes the tendency of the wash liquid to channel or short-circuit and not flush the surface of all the crystals.

Continuous or automatic basket centrifuges are particularly well adapted to the handling of crystals. The economies

of their use are favored by high concentration of crystals in the feed slurry and a mother liquor of moderately low viscosity. Their limitation is the necessity of the solid phases having a well-defined structure of sufficient mechanical strength so that there will be no deformation of the individual particles when subjected to centrifugal force. The ideal material is cubical particles of uniform size, but solids showing substantial deviation from this form are handled successfully. In effect, the requirement is that when the solids are packed under centrifugal force, there will be ample connected void space left in the interstices between the individual particles for the free drainage of the mother liquor. When the particle-size distribution is rather wide, there is a tendency for the smaller particles to fill the voids between the larger ones and offer restriction to the outward flow of the mother and wash liquors.

In the hypothetical plant illustrated in Figure 4, it was assumed that the caustic concentration would be carried to between 15 and 25% as sodium hydroxide in the first-effect mother liquor and between 35 and 40% as sodium hydroxide in the second-effect mother liquor. The sodium chloride concentration would be increased in proportion to the limit of saturation and then crystallization of the excess sodium chloride would occur.

All other factors being equal, larger crystals will be crystallized out from the thinner first-effect mother liquor and more time will be required to spin the more viscous higher concentration second-effect liquor off the crystals. These points are supported by the performance

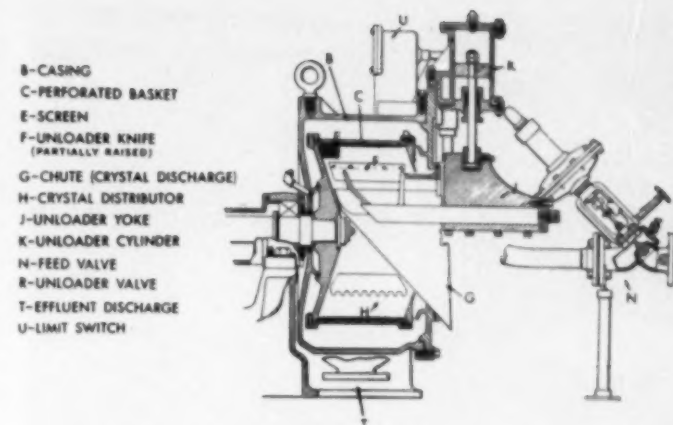


Fig. 5. Cross section of Sharples Super-D-Hydrator.

data of an automatic centrifuge on this application. (See Table I.)

The mother liquor leaving the second-effect centrifuge will be saturated with sodium chloride to the limit of its solubility in the partially concentrated caustic liquor at the temperature of operation. Subsequent evaporation of this mother liquor to the 50% NaOH content, at which it is normally shipped, and cooling to about 90° F. will cause an additional quantity of sodium chloride to crystallize out. Removal of these crystals at this stage cannot be economically performed on a perforate basket centrifuge. The high viscosity of the mother liquor, the small and irregular size of the salt crystals, and their

low concentration are the controlling factors.

In spite of the relatively small size of the dispersed particles of salt, their high density, 2.16, with respect to that of 50% caustic, 1.52, suggests the applicability of a continuous centrifuge of the settling-machine type. Because of the difficulty of making accurate particle-size determinations in such a system, the empirical approach was used. It was determined that a continuous clarifier of the type shown in Figure 8 had good operating characteristics. At a feed rate of 1500 gal./hr. of 50% caustic at between 80° and 85° F., containing not more than 5% of crystallized sodium chloride, an effluent was secured contain-

TABLE I.—DATA ON CRYSTAL SIZE

| Screen Mesh | First-Effect Operation % | Second-Effect Operation % |
|-------------|--------------------------|---------------------------|
| + 10 | 0.0 | 0.0 |
| 10 X 20 | 2.5 | 1.2 |
| 20 X 40 | 10.0 | 8.5 |
| 40 X 60 | 50.1 | 41.9 |
| 60 X 100 | 28.1 | 16.1 |
| 100 X 150 | 9.1 | 12.6 |
| 150 X 200 | 0.0 | 19.7 |

Typical Centrifuge Performance Data

| Operating Cycle | First-Effect Salt Seconds | Second-Effect Salt Seconds |
|-----------------|---------------------------|----------------------------|
| Screen rinse | 1 | 2 |
| Feed | 10 | 15 |
| Cake rinse | 5 | 4 |
| Spin | 20 | 20 |
| Unload | 6 | 6 |

| Operating Cycle | First-Effect Salt lb. | Second-Effect Salt lb. |
|---|-----------------------|------------------------|
| Load/cycle | 60 | 50 |
| Discharged at | | |
| % Water | 2.0 | 2.7 |
| % Sodium hydroxide | 0.15 | 0.40 |
| Using pounds of rinse water/lb. of salt.. | 0.13 | 0.18 |

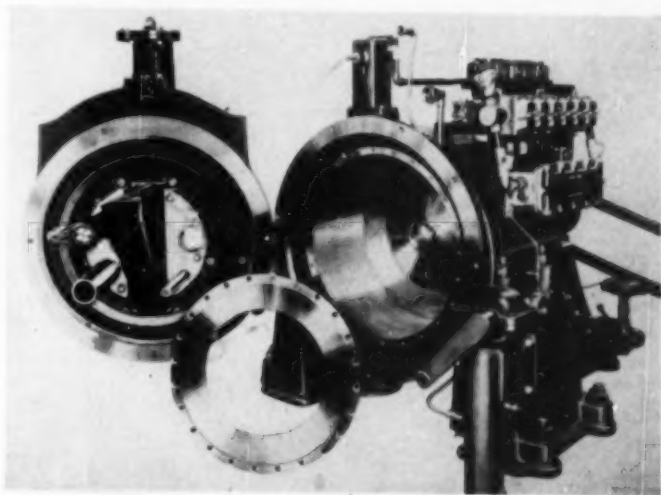
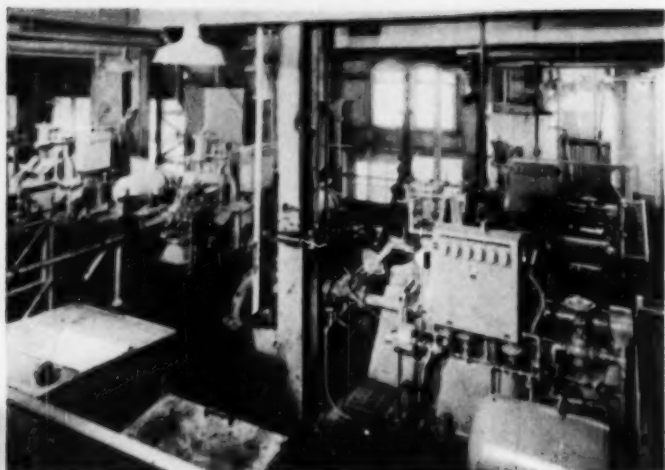


Fig. 6. Exploded view of Super-D-Hydrator.



(Courtesy Pennsylvania Salt Mfg. Co.)

Fig. 7. Centrifuge installation—electrolytic salt.

ing less than 0.1% of residual sodium chloride above its solubility of approximately 1.1%. Up to 1440 lb./hr. of removed salt was discharged at 17% sodium hydroxide and 17% moisture content for recirculation back to the evaporator system.

In this type of centrifuge, the solids are deposited against the bowl wall by sedimentation from a continuous flow of the feed liquid. The solids are conveyed up the slope of the bowl and out of the liquid layer by a helical platter and continuously discharged from the end of the bowl opposite the liquid discharge

ports. While the solids are being conveyed up the dry or "beach" section of the bowl, they are being supported by the plow, and conditions comparable with those of a centrifugal filter exist. As a result, a relatively dry-solids discharge may be obtained from this type of centrifuge when the packed solids exhibit a reasonable degree of porosity. Application of a rinse liquor will remove some adhering mother liquor from such a porous cake, although generally speaking the rinsing efficiency of this type of centrifuge is low because the cake layer that is being washed is essentially tri-

angular in cross section and does not lend itself to uniform penetration by the wash liquid.

Centrifuges of this type are advantageously used for the removal of relatively fine solids from suspension and are often used for handling even relatively coarse suspensions when washing efficiency is not a controlling factor.

Many electrolytic caustic salt plants have found it economically advantageous to convert at least a portion of their chlorine production to organic chlorine derivatives such as paradichlorobenzene, monochloroacetic acid and hexachlorocyclohexane.

In the production of each of these, the basic problem of removing mother liquor from a crystalline material occurs. Centrifuges of the perforate basket type are commonly used in processing the first two.

The centrifuging of paradichlorobenzene introduces a phenomenon of general interest. It is crystallized from a mixture of isomers at approximately 10° C. by careful control of the temperature gradient and of the degree of agitation. When the resultant slurry is centrifuged in commercial equipment, the crystals retained in the basket after equilibrium has been reached have a purity corresponding to a melting point of approximately 50° C. This melting point can be raised above the desired minimum of 52.5° C. with a solvent rinse. However, the introduction of a suitable solvent materially complicates the redistillation of the mother liquor, a step which is necessary for the further recovery of its paradichlorobenzene content.

The desired results can be obtained by elevating the temperature of the crystals in the centrifuge basket. This treatment, in effect, liquefies the surface layer which contains a high concentration of impurities and permits its discharge as a liquid under centrifugal force, leaving the balance of the crystals in the basket at the desired purity. In the case of paradichlorobenzene, rinsing with water at $50.0 \pm 0.5^\circ \text{C.}$ gives entirely satisfactory results with minimum loss of product to mother liquor. The wash water used is easily separated from the mother liquor in a simple gravity tank.

This same principle, again using warm water, has also found wide application in the purification of various naphthalene-bearing stocks from coal-tar fractionation.

Automatic perforate basket centrifuges have been applied also to the purification of low melting point crystallized hydrocarbons. In at least one case it was determined that the required purity could be obtained by rinsing the cake with a recycle stream of the original mother

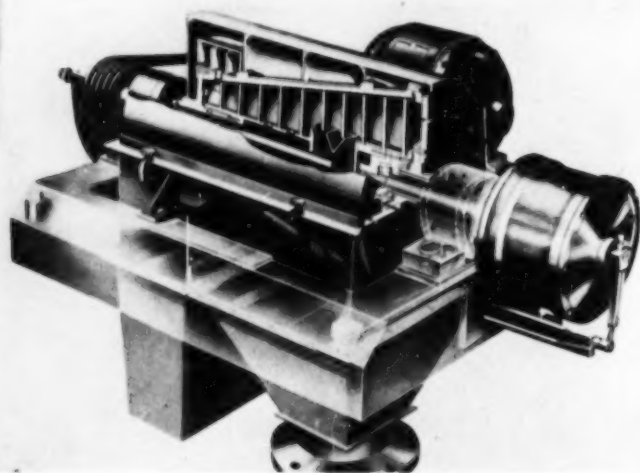


Fig. 8. Phantom view of Sharples Super-D-Canter.

liquor that had been warmed to a suitable temperature differential. Advantages of this from the viewpoint of subsequent processing are obvious.

In contrast to these applications, monochloroacetic acid is usually crystallized from acetic acid mother liquor and pure acetic acid can be used as the rinse liquor without complicating the recycle problem.

Centrifuges of this type are frequently equipped with automatic diversion valves in the effluent line which effect a fair degree of segregation of the rinse liquors, and these are frequently used to advantage in conjunction with multiple-effect evaporators. The relatively weak rinse liquor is returned to the first effect, while the concentrated mother liquor passes on to process.

In the production of hexachlorocyclohexane, a somewhat different problem arises. The active gamma isomer has the greatest solubility in the benzene mother liquor and in order to effect its concentration, the relatively insoluble alpha and beta isomers must be removed from the system. While in some cases these inactive isomers have been secured in a crystal form suitable for processing on a perforate basket centrifuge, the general condition is that they are microcrystalline in size, and the continuous decanter centrifuge gives better operation. In a typical example using a centrifuge of the type illustrated in Figure 8, the alpha and beta crystals are removed from the slurry at a feed rate of 250 gal./hr. and discharged at a residual benzene content of 10% to 12%. For further recovery of its entrained active gamma isomer, this primary cake is repulped in benzene and recentrifuged. The insoluble particles of the inactive isomers do not lend themselves to effective washing in this type of centrifuge. The clarified primary effluent is concentrated by evaporation to yield a 40% active product that may be further purified by additional stages of solvent extraction.

Examples cited have been selected to show some basic differences between applications suitable for processing on the centrifugal-filter and on the settling-machine types of centrifuges. It may be noted that the centrifuges selected for comparison develop approximately the same centrifugal force, in the range of 1000 to 2000 times the force of gravity.

In the use of the other principal product of the chlorine-alkali industry, there are two outstanding examples that illustrate the advantages of high centrifugal force over gravity settling. Ever since soap was first produced by the reaction of wood ashes with tallow, soapmaking has been an art. The soap kettles have become larger (Fig. 9), the chemical and physical-chemical principles have been

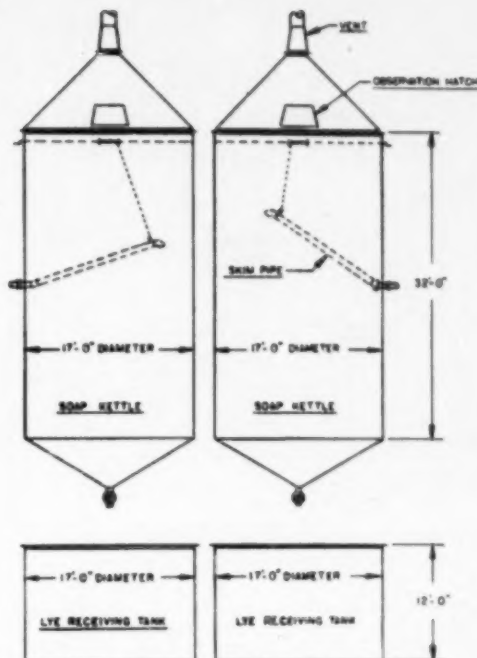


Fig. 9. Soap process kettles.

established, wood ashes have been replaced with sodium hydroxide and home-rendered tallow has given way to a variety of fat formulations for specific end products. Nevertheless, most of the soap, until recently, has been produced by the full boil-kettle process by master soap-boilers, who depend on their senses of taste, hearing, sight and feeling to judge the completeness of the various reactions involved.

Process of Soapmaking

There are four basic steps in the full boil process of soapmaking, and in the language of the art, these are known as changes:

1. Saponification change—Saponification of the fat with sodium hydroxide solution to yield soap and glycerine
2. Strengthening change—Secondary treatment with lye to complete the saponification

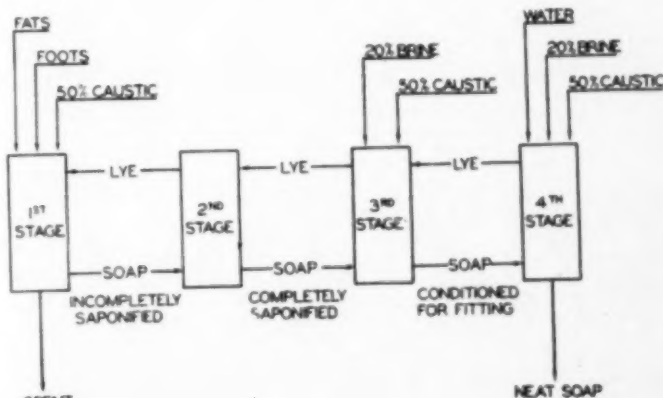


Fig. 10. Flow diagram of continuous soap process.

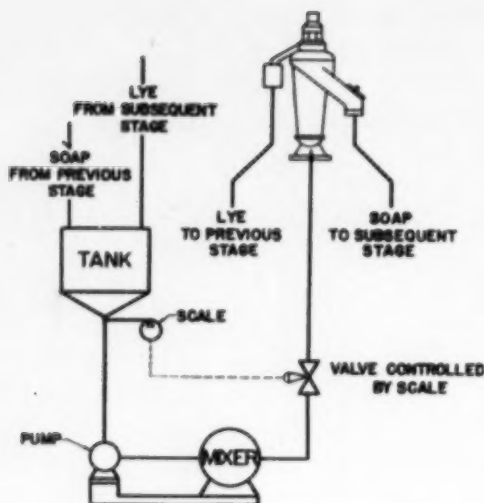


Fig. 11. One-stage continuous soap process.

3. One or more weak changes to wash the soap and adjust the electrolyte content of the charge to prepare it for finishing
4. Fitting or finishing change—the addition of water and electrolyte to the soap, which, up to this point, has been in grain form, in a discontinuous phase, to create a mixture that will separate into melted neat soap as the light phase under the action of gravity and concentrate the remaining impurities and color bodies into a heavy nigre phase

Over and above the time required for reaction and mixing in the large kettles, an additional 16 to 48 hrs. for gravity settling are required in each stage. The electrolyte concentration must be carefully adjusted to maintain the soap in an open or grained condition until the final fitting operation, in order to permit complete separation of the glycerine-bearing spent lye. Even under the most careful operating conditions, spent lye seldom contains more than 5% of

glycerine, and large quantities of water must be evaporated and large amounts of salt, 0.25 lb./lb. or more of fat processed, handled through the glycerine-recovery evaporators and centrifuges.

A continuous process employing the principles of the full-boil soap process and utilizing continuous mechanical mixing instead of agitation in bulk and high centrifugal force for accomplishing the required separation is illustrated in Figure 10. This process operates using conventional fats and oils and caustic soda as raw materials and delivers neat soap and spent lye as end products, the latter up to 24% glycerine content. The amount of salt cycled in process to maintain the required electrolyte concentration is less than 1/5 that required in the kettle process. The flow through the continuous process is divided into four stages, corresponding essentially to the changes of the kettle process, except that the flow of lye is countercurrent to the flow of fat. In mechanical principle, the individual stages are almost identical, a typical one being shown in Figure 11. Each contains the elements of a proportioning device, controls, pumps, surge tank, mixer and centrifugal separator. This process functions under automatic control, and an ordinary operator is easily trained to replace the master soap-boiler. Soapmaking has been elevated from an art to chemical engineering science.

The heart of this continuous soap process is the supercentrifuge in which is developed 13,200 times the force of gravity. In it the grained soap of the first three stages and the melted neat soap of the fourth stage are quickly and completely separated from their respective spent lyes. Processing time is reduced from an average of five days to less than two hours and the plant is much smaller in size. Direct results of the immediate separation and shortened reaction time relative to the conventional kettle process are: (1) reduction in amount of process water, which in turn results in substantially less evaporation load and less salt handling in glycerine-recovery system, and (2) less degradation of soap color and soap quality.

Not only does the neat soap from the continuous process have a better color than that from the kettle, but in the continuous process the fourth-stage nigre is recycled back as third-stage reagent instead of being degraded to a lower grade of soap as is normal in the kettle process.

Figures 12 and 13 show an installed continuous soap plant of a size to convert continuously 1500 lb. of fat/hr. to soap. In this installation, the centrifuges, mixers, and proportioning device

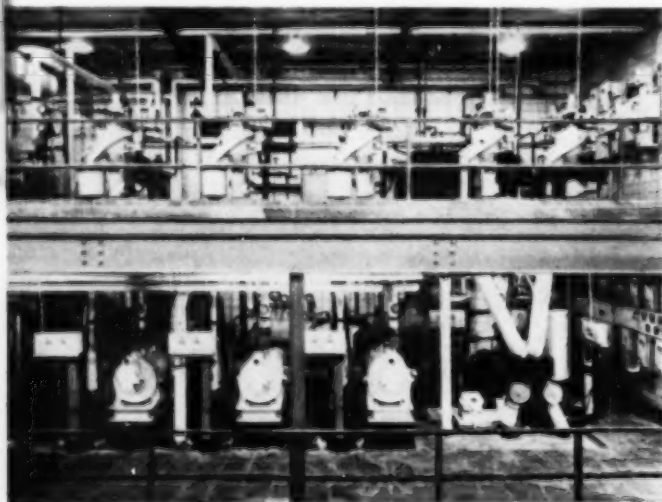


Fig. 12. Installation of continuous soap process. (centrifuges and mixers)

are grouped to facilitate operation. The fat and reagent storage tanks are in the background, and the entire process is operated from a master-control panel.

Another important use of caustic soda is in the refining of crude vegetable oils. The value of olive oil lies in its characteristic taste and aroma. In this, it is practically unique and the market for cooking oil and shortening demands a neutral, bland product of minimum color and having good keeping quality. Before oils such as those recovered from cottonseed, corn, soybeans and peanuts can be decolorized and deodorized by physical-chemical means, they must be refined. In the crude form these oils contain as impurities vegetable gums, certain phospho-lipoids, free fatty acid and color bodies, all of which react with aqueous alkaline solutions, in varying degrees, to form oil insoluble products. Unfortunately, these reaction products, the hydrated gums and soap, also adsorb the desired end product, neutral oil, and, furthermore, in the presence of soap, the neutral oil is rather easily saponified under the conditions of elevated temperature and caustic concentration necessary to remove the color bodies. When the refining is carried on in kettles with gravity settling of the reaction products, the settled soap stock may retain upward of 30% of neutral oil; or to express it another way, 50% to 60% of the total fat content of the soap stock will be neutral oil. Almost all vegetable oil is now refined continuously with centrifugal separation of the oil and soap stock. The original process employing caustic soda as the reagent substituted continuous mixing, heating and centrifugal separation for the refining kettle. Yields are improved, primarily because of the ability of centrifugal force to squeeze more oil out of the soap stock.

For instance, on several comparative runs that gave soap stock containing 50% to 60% of neutral oil on the basis of the total fat content by kettle refining, centrifugal separation gave the following values:

| Type of Oil | NEUTRAL OIL IN SOAP STOCK AS % OF TOTAL FAT | |
|-------------|--|-----------------------|
| | @ 6000 X Gravity | @ 13,200 X Gravity |
| Peanut A | 34.4 | 27.0 |
| Peanut B | 32.8 | 24.2 |
| Cotton | 43.0 | 37.9 |
| Corn | 45.7 | 40.0 |
| Soya | 41.0 | 37.9 |

In an improved multistage process, the gums are first hydrated with a weak alkali, sodium carbonate solution, and then fixed or caused to lose their adsorptive properties by a dehydration step. Sufficient additional sodium carbonate is then added to neutralize the fatty acid content of the oil and plasticize the gums

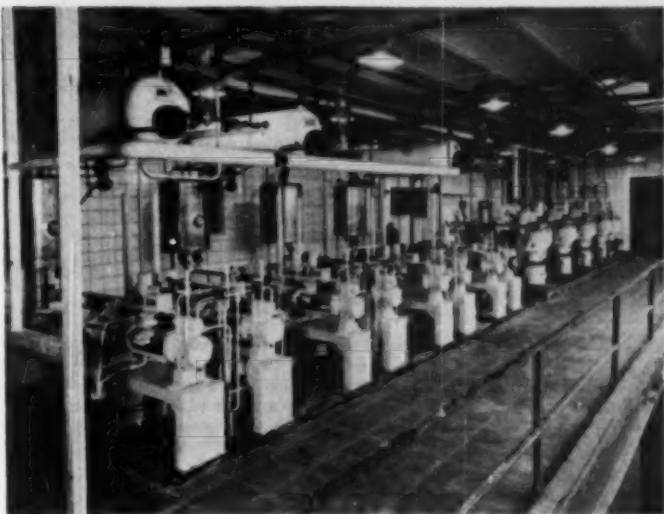


Fig. 13. Installation of continuous soap process.
(proportioning pump and centrifuges)

so that the reaction products can be centrifugally separated continuously. Because of the use of the weaker alkali, saponification does not occur to any measurable extent and the loss of neutral oil is almost entirely due to entrainment. A secondary treatment of the centrifugally separated oil with strong caustic is required for color reduction, but little loss takes place at this point because there is practically no soap present to promote saponification, the free fatty acid having been neutralized in the preceding stage. This refining method gives neutral oil yields within 1% or 2% of theoretical, a more than acceptable value for most organic reactions. Even this loss is sufficiently high to encourage further development work.

Recent experimental runs under conditions that gave no saponification have reduced the oil loss by entrainment in the soap stock to as little as 18% of the total fat content.

The centrifuge has become a valuable tool of the process industries. Its use appears to be limited only by the skill of the chemical engineer who must adapt process problems to reap the fullest advantage and benefits of centrifugal force.

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(Presented at Thirteenth Regional Meeting, Houston, Tex.)

CATALYTIC REACTIONS AT HIGH PRESSURES AND TEMPERATURES†

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The purpose of this investigation was to design and construct a continuous, small-scale plant for studying catalytic reactions at elevated temperatures and pressures. Continuous operation of this plant at 1200° F. and 10,000 lb./sq.in. confirmed the applicability of this equipment for studying chemical reactions over a wide range of pressures, temperatures, and flow rates.

In order to demonstrate the operation of this equipment, the decomposition of butanol-1 over an alumina-silicate catalyst was investigated at 760° F., at pressures of 1 atm. 750, 2500, and 9000 lb./sq.in., and a feed rate of 0.2 to 1.5 lb. moles of feed/(hr.) (lb. of catalyst).

It was found that at a given rate of feed, the decomposition of butanol-1 first increases with increasing pressures and thereafter decreases with increasing pressures. The exact pressure at which maximum decomposition occurs was not determined. At 2500 lb./sq.in. the decomposition is less than at 750 lb./sq.in. but greater than at atmospheric pressure. The decomposition at 9000 lb./sq.in. is less than at atmospheric pressure.

Although all the products of reaction were not identified, the results indicate that the major decomposition reaction is the direct dehydration of butanol-1 to mixed butenes and water. Complex side reactions, involving probably only the butenes also occur; the extent of these side reactions increases with increasing pressures. The major side reactions involve the condensation of the mixed butenes to higher molecular weight substances.

THE purpose of this work was to design and construct a continuous small-scale plant for studying catalytic reactions at temperatures as high as 1200° F. up to pressures of 10,000 lb./sq.in. Although similar equipment has been used in the past for studies of this type (14), there is little information in the literature on chemical reactions for pressures above 5000 lb./sq.in. except where the temperatures are kept well below 1000° F. (8). Above 1000° F., where "creep" and possibly hydrogen attack become serious, the problems involved in the mechanical design of the equipment are complex (18).

This plant has been operated continuously at 1200° F. and 10,000 lb./sq.in. using water and butanol-1. At a lower temperature, around 1000° F., it is estimated that the permissible operating pressure would be greater than 20,000 lb./sq.in. Because of the satis-

factory results that were obtained at these extreme conditions, particularly the high temperature, it is believed that the present design is somewhat unique in this respect.

In order to demonstrate the operation of the plant, the catalytic decomposition of butanol-1 at elevated temperatures and pressures was investigated.

Description of Equipment

A schematic flow diagram of the equipment is shown in Figure 1. Two 500-ml. graduated cylinders (1), connected by a three-way stopcock act as feed reservoirs for the dual feed, plunger-type, Hills-McCanna pump (3). The pump is equipped with suction risers (2), and discharge risers (4), which serve to prevent vapor-lock or air binding in the pump cylinder. Two Amino rupture disc assemblies (5), are located immediately downstream from the pump and are vented to the central venting system which exhausts to the outdoors (27).

Two 30-ft. coils of stabilized stainless steel tubing, Type 321, 1/4-in. O.D. by 0.085-in. I.D., are located in an Eclipse pressed steel pot which serves as a preheater and is filled with Du Pont heat treating salt No. 1. The pot is heated by means of chromel resistance wire embedded in an alundum core (7).

The two feeds from the heater, then fed to the reactor, are purposely not allowed to mix until they reach the reactor in order to minimize thermal, noncatalytic reactions in the preheater.

The reactor (9) is machined from a forging of Uniloy 19-9 WMo (highly alloyed stainless steel) and is about 3 ft. long. The I.D. of the reactor is 3/4-in. and its O.D. is 2 1/2 in. The reactor is equipped with a Black, Sivalis, and Bryson special safety head of stainless steel (8) and is also vented to the central vent line (27). Three separate radiant furnaces serve as reactor heaters (10). Each heater consists of chromel wire embedded in an alundum cement core. The heat input to each heater unit is controlled separately by means of three Adjustavolts.

The bottom of the reactor is fitted with a 1/4-in. O.D. x 3/16-in. I.D. stainless steel thermocouple tube (13), which extends the whole length of the reactor. A traveling thermocouple wire embedded in a two-holed ceramic insulator, permits measurement of the temperature over the full length of the reactor.

The salt pot and reactor furnaces are enclosed in a single housing. Firebrick and silocel are used as insulation. Each heater unit and the salt bath are equipped with chromel-alumel thermocouples.

The outlet from the bottom of the reactor includes a pressure gage connection (12) and a connection to the condenser (15). The condenser consists of coiled stabilized stainless steel tubing, 1/4-in. O.D. x 0.085-in. I.D. and a water box cooler. The condenser line extends to a filter (16), and a stainless steel Amino needle valve (17) by means of which the pressure on the entire apparatus is controlled.

Copper tubing, 1/4-in. O.D., connects the valve to the sampling manifold which is provided with proper by-passes of rubber tubing, brass valves, and glass stopcocks in order to render a completely enclosed sampling system. In normal operation the discharge from the condenser passes to two vertical glass condensers and then to two liquid-product, graduated, receivers (19). The gas passes overhead from these two 500-ml. receivers to an entrainment separator (20). The gas then is metered in a Sargent wet-test meter (21), and exhausts through the central venting system (27).

Provision is also made for periodically drawing off a gas sample through a boric acid scrubber (22), and a drying tube (23), to the sampler (24). A standard 87% H₂SO₄ Orsat analyzer is also provided (25).

The part of the apparatus included between the discharge risers (4), and the sampling manifold (18), is located within an enclosure measuring 5 ft. x 6 ft. and 7 1/2 ft. high. The enclosure consists of 1/2-in. steel plate welded on a 1/4-in. by 1 1/2-in.

† Complete original data in Tables 1, 2, and 3 are on file (Document 2930) with American Documentation Institute, 1719 N. St. Northwest, Washington, D. C. Microfilm obtainable by remitting 50 cents and 50 cents for photoprints.

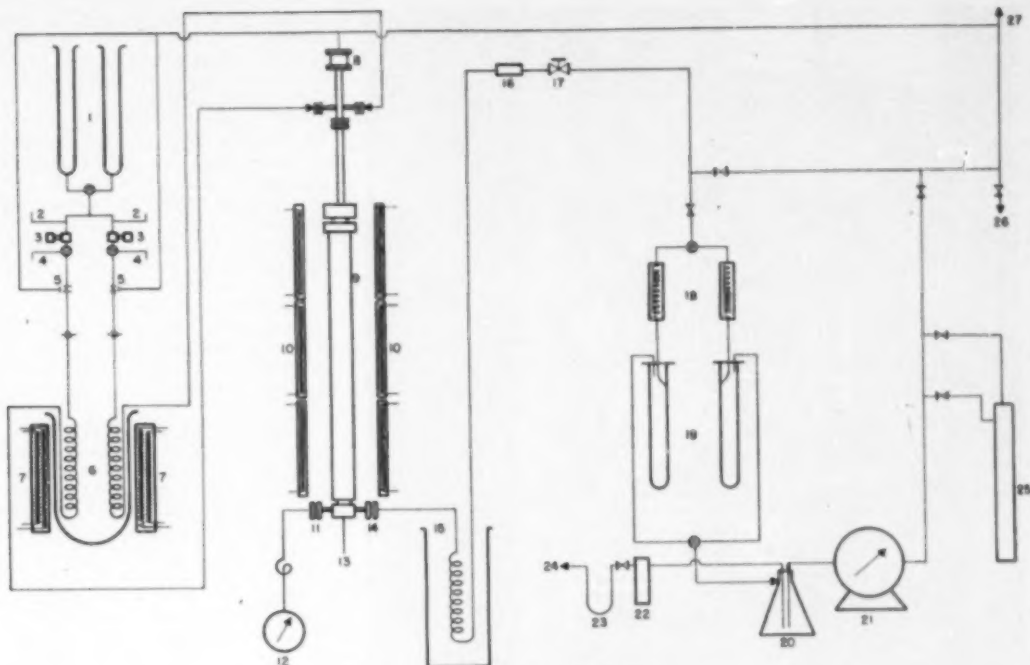


Fig. 1. Schematic flow sheet of equipment.

angle iron framework and is equipped with a $\frac{1}{2}$ -in. steel door.

Figure 2 shows a front view of the panel board, pump, and the sampling manifold on the right. In the center of the photograph and immediately above the pump is a calibrated dial and a handwheel. The handwheel is connected to a shaft that extends through the steel enclosure to one of two pulley wheels, which are attached to the thermocouple tube extending from the bottom of the reactor. This arrangement serves as a mechanism for moving a thermocouple along the full length of the reactor.

Decomposition of Butanol-1

In order to demonstrate the utility of the equipment described above a study of the decomposition of butanol-1 at elevated pressures and temperatures was undertaken. This particular reaction was selected as the result of a recommendation by the War Production Board, which was at that time interested in dehydration and hydration studies.

The catalytic decomposition of butanol-1 was first accomplished by LeBel and Green in 1881 in the presence of hot ZnCl_2 (24). Although many investigators have studied the decomposition of butanol-1 since then, none has reported the effect of elevated pressures in flow systems on the decomposition reaction. Decomposition at elevated pressures of ethyl alcohol (10) and isopropyl alcohol (13) was studied in batch systems.

In the present work the following were investigated: effect of pressure and space velocity (feed rate) on the decomposition of butanol-1 over an alumina-silicate catalyst in the temperature range of 750-770° F., a pressure range of 1 atm. to 9000 lb./sq.in., and a feed rate of from 0.2 to 1.5 lb. moles of feed/(hr.)(lb. of catalyst). This temperature range was selected because it permitted the study of a wide range of dehydration values in this particular equipment without favoring the predominance of side reactions (18).

Materials. Normal butanol (b.p. 115-118° C., flash point 116° C., 0.005 cc. left when 100 cc. evap. to constant weight) was obtained from the Commercial Solvents Corp. The bead catalyst (4-10 mesh) was supplied gratis by the Socony Vacuum Oil Co., Detroit, Mich. The catalyst is similar to the one used in its cracking operations. It consists of 10-15% alumina and the remainder silicate. Sodium silicate and sodium aluminate are the only materials used in its manufacture.

Operating Procedure. On the basis of a series of preliminary runs (18), the following procedure was adopted.

A mixture of 50 g. of bead catalyst and 80 g. of zirconite chips was placed in the reactor to a depth of about 17 in. The pur-

pose of the zirconite chips was to dilute the catalyst and thereby facilitate more uniform temperature regulation over the catalyst bed during the endothermic reaction. The average temperature variation over the entire catalyst bed at any instant did not exceed 10° F.

The reactor closures and connecting lines were then assembled and the reactor was brought to temperature in about 5 hrs. Once the desired temperatures—as estimated from the data on the preliminary runs (18)—were established throughout the system, freshly boiled (air-free) *n*-butanol was passed continuously over the catalyst at a predetermined rate for approximately 80 min. before any samples were taken. During the preliminary runs it was found that when butanol was first introduced over freshly regenerated catalyst, the catalyst displayed high initial activity. After about a half-hour, the catalyst activity and temperature became stabilized, as did the rate of formation of gaseous products. For approximately another hour, the gas rate remained relatively constant before a gradual decrease was observed.

During the process period the gas product rate in seconds/0.1 cu.ft., the feed rate, and the liquid product rate were recorded at 10-15-min. intervals. During this period the pressure and feed rate were maintained constant. The temperatures along the reactor bed were recorded also by means of the traveling thermocouple and were averaged to obtain the reaction temperature. At the end of the process period a sample of the liquid product (about 250 cc.) and two samples of the gas product were taken simultaneously. One of the gas samples was directly withdrawn into the Orsat apparatus attached to the equipment, and another was withdrawn into an evacuated

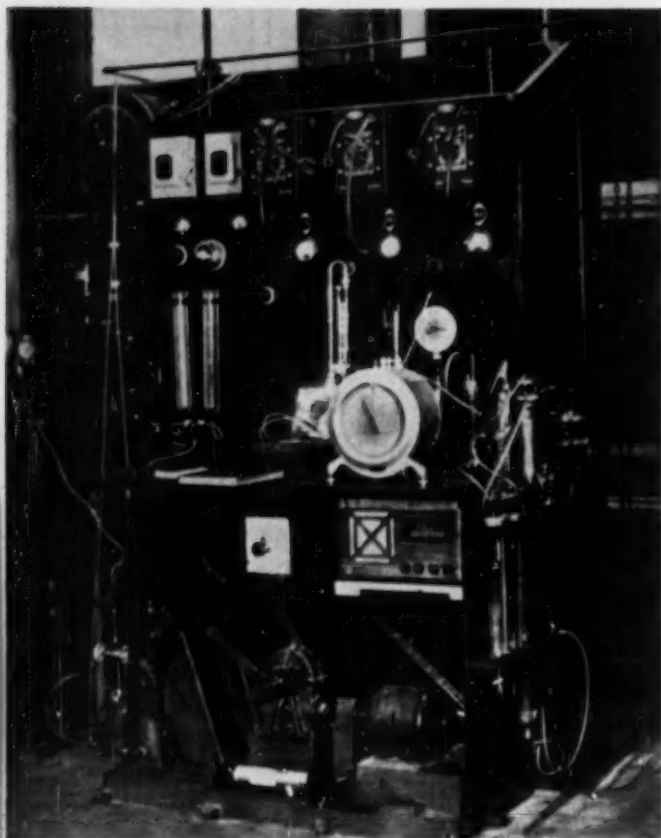


Fig. 2. Panel board and pump.

was bulb for subsequent analysis in the infrared spectrometer.

At the conclusion of each process period the butanol feed was discontinued and the catalyst was flushed with steam to remove any oily polymers. Air was then passed over the catalyst at a slow enough feed rate so that the temperature did not rise more than 50 degrees due to the combustion of the carbonaceous deposit on the catalyst. After about 10 min. on air stream, the system was thoroughly purged with nitrogen, and a new process period was begun.

Runs were made at 750° F. and pressures of 1 atm., 750, 2500, and 9000 lb./sq.in. at four space velocities. Frequent check runs were made to determine whether the catalyst activity had been reproduced after regeneration.

Analysis of Products. The analytical work consisted primarily in the chemical determination of the unreacted alcohol and water in the liquid product samples, and the analysis of each gas sample by the Orsat and the infrared spectrometer. Several other miscellaneous tests were made.

Each gas sample was analyzed in a standard Orsat apparatus equipped with

an absorption bulb containing 87% H_2SO_4 . In most cases the absorption was greater than 98% (unsaturated butenes). Each gas sample was also examined by means of an infrared spectrometer, using both the NaCl and KBr prisms. Since the quantitative determination of the constituents in the complex gaseous mixtures would have involved the determination of many calibration curves, the method of analysis was used purely for a qualitative comparison of the various gas samples. In general, all the absorption curves were practically identical. Through the courtesy of The M. W. Kellogg Co., Jersey City, N. J., several gas samples taken under widely varying conditions of pressure and space velocity were analyzed in a mass spectrometer. Results showed only minor variations. The composition reported for Series No. 8, which is representative, is as shown in Table A.

All the liquid products collected from each series were analyzed for water and

alcohol. In most cases the liquid samples consisted of two layers; therefore, separate determinations were made on each layer. The specific gravity of each layer was also recorded.

The amount of alcohol was determined by means of a modification (20) of the standard potassium dichromate oxidation of the alcohol to an acid (22). The accuracy of this method is well within $\pm 1\%$.

The amount of water was determined by means of the Karl Fischer method (19).

The quantity of carbon deposited on the catalyst was determined by passing air over used catalyst and analyzing for carbon dioxide and carbon monoxide in a standard KOH and Cu_2Cl_2 absorption train.

Various miscellaneous tests on the upper liquid layer indicated that only traces of aldehydes were present. Because of analytical difficulties, no direct tests for ethers were made. However, it will be shown later that the quantity of ethers formed is negligible.

With increasing pressures the color of the upper liquid layer gradually changed from a slight yellowish tint to a deep reddish brown. Table 1 summarizes the effect of progressively increasing pressures at 760° F. on the coloration of the liquid products (upper layer) from the catalytic decomposition of butanol-1.

Results

Since the liquid samples were taken at the termination of the process period when the temperature was not necessarily 760° F. (corrected) the extent of the alcohol decomposition, as determined from the chemical analysis of the liquid samples, also had to be corrected to the datum temperature of 760° F. During the course of the process period the average temperature in the reactor bed varied from 15 to 20° F. As shown in Figure 3 (which is typical of the runs at the other pressures) the moles of wet gas produced per mole of feed is a linear function of temperature. By assuming that the ratio of the moles of

TABLE A

| Constituent | Mole % |
|------------------------------------|-------------------|
| $\text{N}_2 + \text{CO}$ | 0 |
| Hydrogen | 0.6 |
| Methane | 0 |
| Ethylene | Trace |
| Ethane | 0 |
| Propane | 0 |
| Butadiene | Possible Trace |
| Isobutene | 3.2 |
| Butene-1 | 28.7 |
| Butene-2 | 67.0 |
| n-Butane | 0 |
| C ₆ 's and heavier | 0.5 |

wet gas produced to the moles of alcohol decomposed is a constant over this small temperature interval (15-20° F.) for a particular feed rate and pressure, it was possible to correct the alcohol determinations obtained from the liquid samples to a common datum of 760° F. To verify this assumption, two liquid samples were taken at different average catalyst temperatures in the runs of Series No. 10. The ratio of the moles of wet gas produced to moles of alcohol decomposed was 0.99 at 761° F. and 0.973 at 749° F. These results are well within the experimental error.

The actual experimental data (18) from each series of runs were corrected to a datum temperature of 760° F. by the method described above and are summarized in Table 2 and in Figure 4. In Figure 4 the term W/F is expressed in mass of catalyst per mole of feed per hour, and it is related to the reciprocal of the space velocity. Because of insufficient data at low conversions (small values of W/F) the shape of the curves in this region was established by means of an empirical correlation based on pseudo-first-order kinetics (9). Thermodynamic calculations (21) on the dehydration of butanol-1 at 760° F. reveal that at equilibrium, the alcohol is practically 100% decomposed, even at 9000 lb./sq.in. Therefore, the curves of Figure 4 should approach 100% conversion as W/F approaches infinity (equilibrium). For this reason, the computed point at

$$W/F = 2.9 \frac{(\text{lb. catalyst}) (\text{hr.})}{(\text{lb. moles feed})}$$

and at atmospheric pressure is assumed to be in error since it is improbable (but not impossible) that this curve would level off in this region and then proceed to rise asymptotically to the 100% conversion curves.

Possible Chemical Reactions. If

TABLE 1

PHYSICAL PROPERTIES OF THE UPPER LAYER OF THE LIQUID PRODUCT FROM SERIES NO. 14

| Sample | $\frac{W}{F}$ in (lb. catalyst)(hr.) lb. moles feed | Gauge Pressure lb./sq.in. | Specific Gravity 22°C | Refractive Index 25°C | Color Index* Percent Light Transmission |
|--------|---|---------------------------------|-----------------------------|-----------------------------|--|
| 14A | 1.46 | 0 | 0.840 | 1.3917 | 97.1 |
| 14B | 1.46 | 1500 | 0.818 | 1.3942 | 89.7 |
| 14C | 1.45 | 3000 | 0.812 | 1.3946 | 84.8 |
| 14D | 1.46 | 4500 | 0.810 | 1.3993 | 80.4 |
| 14E | 1.37 | 6000 | 0.797 | 1.4030 | 69.1 |
| 14F | 1.39 | 7500 | 0.778 | 1.4032 | 33.1 |
| 14G | 1.53 | 9000 | 0.783 | 1.4031 | 30.3 |
| 14H | 2.74 | 9000 | 0.878 | 1.4343 | 21.6 |

*Note: Relative color index based on pure n-butanol having 100 per cent light transmission. Instrument: Fischer Colorimeter, Green (610Å) Filter

TABLE 2

SUMMARY OF RESULTS OF THE DECOMPOSITION OF N-BUTANOL IN THE PRESENCE OF ALUMINA-SILICATE CATALYST AT 760° F.
(MASS OF CATALYST = 50 gm.)
VOL. OF CATALYST BED = 0.00361 Cu.Ft.)

| Series | Gauge Pressure lb./sq.in. | $\frac{W}{F}$ in (lb. catalyst)(hr.) lb. moles feed | Moles Alc. Decomp. | Moles Butenes Mole Alc. Fed. | Length of Process Period Min. | Esti- mated Contact Time Sec. |
|--------|---------------------------------|---|-----------------------|---------------------------------|--|---|
| 1 | 0 | 0.631 | 0.322 | 0.332 | 80 | 0.0766 |
| 4C | " | 0.945 | 0.417 | 0.370 | 80 | 0.1152 |
| 7 | " | 1.359 | 0.535 | 0.496 | 36 | 0.1510 |
| 14A | " | 1.460 | 0.653 | 0.603 | -- | 0.1552 |
| 15A | " | 1.419 | 0.536 | 0.456 | 80 | 0.1570 |
| 10A | " | 2.90 | 0.566 | 0.533 | 80 | 0.318 |
| 10B | " | 2.90 | 0.597 | 0.533 | -- | 0.319 |
| 2C | 750 | 0.732 | 0.449 | 0.330 | 85 | 4.62 |
| 2 | " | 0.732 | 0.441 | 0.330 | 85 | 4.84 |
| 5 | " | 1.030 | 0.530 | 0.475 | 85 | 6.17 |
| 8 | " | 1.432 | 0.721 | 0.561 | 60 | 8.15 |
| 15B | " | 1.523 | 0.503 | 0.471 | 60 | 9.16 |
| 11 | " | 3.065 | 0.630 | 0.737 | 60 | 16.80 |
| 3 | 2500 | 0.744 | 0.447 | 0.296 | 80 | 18.80 |
| 6 | " | 1.055 | 0.595 | 0.415 | 85 | 22.05 |
| 9 | " | 1.46 | 0.653 | 0.499 | 80 | 34.0 |
| 15C | " | 1.51 | 0.651 | 0.449 | 60 | 38.0 |
| 12 | " | 3.03 | 0.646 | 0.771 | 75 | 65.4 |
| *13C | 9000 | 1.22 | 0.435 | 0.282 | -- | 180.1 |
| *13 | " | 1.29 | 0.450 | 0.282 | -- | 184.9 |
| *14C | " | 1.53 | 0.615 | 0.370 | -- | 184.5 |

* These runs have not been corrected to the 760° F. datum. The values for No. 13C are 763° F., for No. 13 are 761° F., and for No. 14C are 765° F. Because of mechanical difficulties encountered in the operation of the pump at 9000 lb./sq.in. (gm) sufficient data were not obtained to establish the effect of temperature on the conversion. However, the curve at 9000 lb./sq. in. of Fig. 1 was corrected to a 760° F. datum with the help of the temperature-conversion data at 2500 lb./sq. in.

the only reaction taking place were the dehydration reaction,

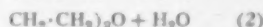


the moles of butanol decomposed would be equal to the moles of butenes formed. Data of Table 2 show that the moles of butenes formed are less than the moles of alcohol decomposed, the difference between them increasing as the pressure increases. Chemical analysis of the liquid products proved that constituents other than water and alcohol were present. Also, the fact that the

hydrocarbon layers of the liquid samples were colored, the coloration increasing with pressure, indicated qualitatively that reactions, involving the formation of higher hydrocarbons, were taking place.

Attempts to identify positively the side products were not successful because of analytical difficulties. Therefore, in order to draw any conclusions on the nature of the side reactions, it was necessary to consider the various other reactions that might take place.

If the only reaction taking place were the dehydration reaction, the moles of butanol decomposed would be equal to the moles of butene produced and to the moles of water produced. Chemical analyses revealed that the moles of butenes produced were less, and that the moles of water produced were more, than the moles of alcohol decomposed in all cases except in the runs at high feed rates and zero pressure, where side reactions are at a minimum. The only other likely reaction which would produce water at these elevated temperatures (15) is given by



In the formation of ether, only one mole of water is produced for every two moles of alcohol decomposed. Therefore it is improbable that the ether reaction

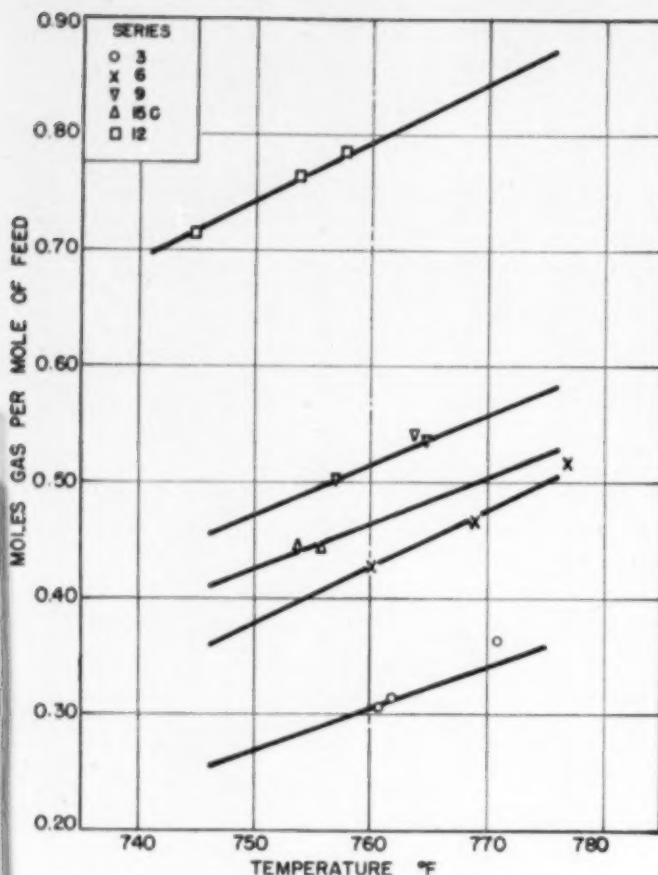
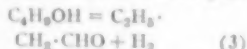


Fig. 3. Effect of temperature on decomposition of butanol-1 at 2500 lb./sq.in. gage.

occurs to any appreciable extent since the moles of water produced were consistently higher than the moles of alcohol decomposed.

If the following reaction takes place



alcohol would be consumed without the formation of water; however, if this reaction occurred at all, the moles of water formed would be less than the moles of alcohol decomposed. Chemical analysis of the liquid products indicated that only a trace of aldehydes was present, and a mass spectrographic analysis revealed that only small amounts of hydrogen were present in the gas.

Another possible mechanism for the formation of the aldehyde is as follows:



Since the mass spectrographic analyses of the gas samples revealed that no butanes were present except for the run at 9000 lb./sq.in., it is doubtful whether this reaction proceeds to any significant extent. At 9000 lb./sq.in. the concentrations of hydrogen and butanes were 4.0 and 3.6 mole % respectively. The runs at 2500 lb./sq.in. and below averaged 0.6 mole % hydrogen and no butanes.

The increased concentration of butanes at 9000 lb./sq.in. may well be due to Reaction 4. The increased hydrogen is probably a combined result of Reaction 3, decomposition or cracking of butenes, and condensation of the unsaturates to form cyclic compounds, such as benzene, toluene, cyclohexenes, cyclohexadienes, etc., and hydrogen (23). Although the analytical methods used were not sufficient to identify these cyclic compounds, there is evidence that these condensation reactions occur. For example, the quantity of "unidentified" constituents in the liquid samples in-

creased with pressure; at 9000 lb./sq.in. they amounted to about 20 wt. % of the total liquid product.

Thermodynamic calculations (21) reveal that at equilibrium Reaction 1 is practically 100% complete; Reaction 2 occurs to a limited extent; Reaction 4 is practically negligible, and Reaction 3 is negligible.

Based on the above tests, which appeared to restrict the decomposition of alcohol to olefin and water, there appeared to be only two explanations to account for the excess water in the liquid product:

1. Since the accuracy of the water determinations by the Karl Fischer method was shown to be high (16), the only likely error could be in the volumetric measurement of the liquid sample. In fact, in some runs, a small error in the reading of the liquid product rate could result in an appreciable error in the moles of water produced since the volume of the water layer (averaging 90 to 95% water) formed was relatively small compared with the hydrocarbon layer (which averaged less than 15% water). However, it is unlikely that errors in reading the liquid product rate would yield consistently high results.

2. Out of 49 runs in which material balances were made on the feed, in, and the product, out, approximately 75% of these runs showed a feed to product ratio of less than one. During regeneration of the catalyst, steam, then air, and finally nitrogen were passed over the catalyst. Any water, which remained in the system (from the initial steam purge) upstream from the point of inlet of the air purge stream, passed over the catalyst with the first alcohol feed of the succeeding run. Since the alumina-silicate catalyst readily adsorbs water, a major portion of the water was probably adsorbed on the freshly regenerated catalyst. During the processing period, this water probably was desorbed gradually and passed out of the reactor with products, which would account for the excess water in the liquid product and the high material balance.

Two runs in which there was a significant excess of product over feed, cited to verify this conclusion, are:

| Series | T °F. | Excess g. H ₂ O | Excess Product |
|--------|-------|-------------------------------|-------------------|
| 2(C) | 757 | 4.2 | 4.4 |
| 3 | 756 | 4.48 | 3.04 |

Therefore, it was concluded that the excess water observed in all runs was due to water adsorbed on the catalyst during the regeneration period and desorbed during the run.

Only one other mechanism is apparently possible for the formation of olefin and water from alcohol. The olefin may be produced through intermediate ether formation which subsequently decomposes into olefin and water. Although Ipatieff (10) observed this phenomenon in the case of ethyl alcohol (which dehydrates at approximately the same rate as ether) Adkins (2) concluded that butenes are not

formed through the intermediate ether step because butyl alcohol dehydrates at a faster rate than butyl ether.

Since all the alcohol that decomposes forms water—which does not further react—and olefin, any side reactions must be attributed to the butenes formed. Aside from isomerization reactions the butenes can undergo two types of reactions: (1) Decomposition into lighter products or (2) Condensation into heavier hydrocarbons. Since the mass spectrograph analysis of the gas does not indicate appreciable amounts of substances lighter than butenes, the major side reaction taking place must then be the condensation reaction. If this is true, then the weight of unidentified constituents in the upper layers* of the liquid products must exactly account for the difference between the grams of butenes actually present in the product and the grams of butenes that would have been formed if only the dehydration of the alcohol took place. To check this assumption the grams of unidentified constituents in the liquid product were added to the grams of butenes in the gaseous product, and the sum was converted to moles of "equivalent butenes." Values for the moles of equivalent butenes formed as compared with the moles of alcohol

* The lower liquid layer consisted almost entirely of water and butanol whereas the upper layer contained the liquid side reaction products.

TABLE 3

PRESSURE - 750 lb./sq. in.

| Run No. | Moles of Alcohol Decomposed | Moles of "Equivalent Butenes" Formed |
|---------|-----------------------------|--------------------------------------|
| 2C | 0.463 | 0.468 |
| 2 | 0.522 | 0.492 |
| 5 | 0.610 | 0.570 |
| 8 | 0.716 | 0.667 |
| 15B | 0.688 | 0.660 |
| 11 | 0.792 | 0.790 |

decomposed are summarized in Table 3 for the runs at 750 lb./sq.in.

Results at other pressures were in similar agreement. Therefore, within the limits of experimental error the hypothesis that only butenes participate in side reactions seems justified.

As to the isomerization reactions, it has been verified by the mass spectrographic analysis and the infrared absorption curves of the gas that all the isomeric forms of butenes are present. Ratios of butene-2 to butene-1 are of the same order as the equilibrium values reported in the literature (12, 17, 20).

The difference between the moles of alcohol decomposed and the moles of butenes actually formed (Table 2) represents the extent of the side reac-

tions measured in terms of equivalent moles of butenes participating in side reactions per mole of butanol fed. From the data of this table it appears that the moles of butenes produced per mole of butanol fed progressively increases with W/F . However, it is conceivable that for high values of W/F the butenes formed will reach a maximum and thereafter decrease with further increases in W/F . This reversal could be attributed to increasing side reactions which consume the butenes at a faster rate. To verify this fact, data at high values of W/F are required. The catalytic dehydrogenation of butanes to butenes and dealkylation products behaves in this manner at temperatures exceeding 1060° F. (5).

Kinetic Analysis. It has been definitely established that the decomposition of alcohol is a heterogeneous reaction (10, 11).

Ipatieff (10) showed that ethanol does not decompose even at 500-520° C. in the absence of a catalyst. Since the rate of dehydration of alcohols increases in the order of *n*-butyl, propyl, isobutyl, ethyl, isopropyl, and secondary butyl alcohols (2), it is highly improbable that *n*-butyl alcohol will undergo homogeneous dehydration at 400° C. (750° F.).

In the present case, the alumina-silicate bead catalyst and the iron walls of the reactor vessel both catalyze the decomposition. However, it is believed that the iron catalysis at the temperatures involved was negligible (11) since iron favors the formation of aldehyde instead of olefin and only a trace, if any, aldehyde was detected in the products. Therefore no attempt was made to elaborate further on the noncatalytic decomposition of *n*-BuOH in this present investigation. The zirconite chips used to dilute the catalyst did not appear to catalyze the decomposition.

A complete kinetic analysis of the decomposition of butanol would require a

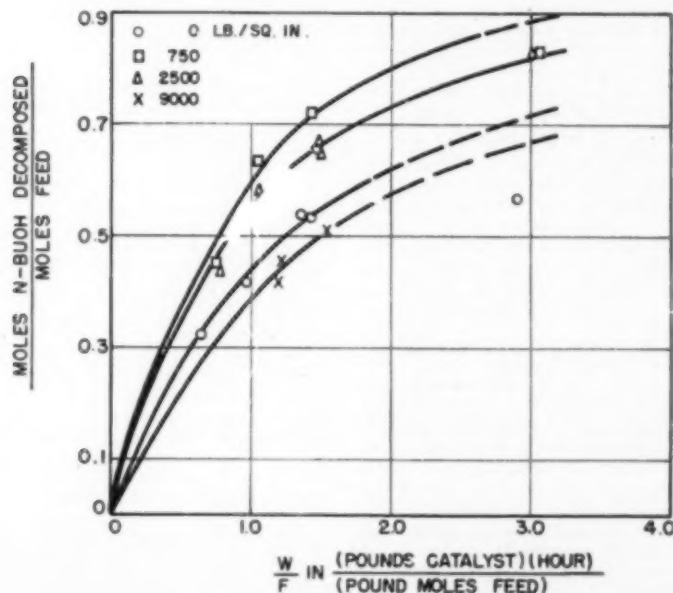


Fig. 4. Decomposition of butanol-1 at 760° F.

precise knowledge of the reactions that take place and the mechanism by which the products are formed. Once the extent and nature of the reactions are established, the general rate equations can be derived (7, 9). Invariably, in complex systems these methods depend on the simultaneous graphical integration of several rate equations. This procedure is not only tedious but also gives no assurance of a unique solution (4).

In all probability the range of conditions that were investigated is too great to permit an over-all correlation. For example, in attempting to predict the apparent order of the over-all decomposition reaction, the log of the apparent time in seconds required to produce 50% decomposition was plotted against the log of the pressure in pounds per square inch. The contact time was calculated on the basis of the arithmetic average volume of the reactants and products. The volumes were corrected for deviations from ideal gas behavior by use of the generalized compressibility factor plot and the pseudocritical concept. Although the points fell along a straight line, the apparent order of the reaction as determined from the equation of this line was found to be -0.3 which is meaningless. Practically the same equation was obtained when the contact times were calculated by the method outlined by Brinkley (3), even though the contact times as computed by the two methods differed as much as 90% at the higher pressures.

Referring to Figure 4 it will be noticed that increasing the pressure to 750 lb./sq.in. materially increased the decomposition but further increases in pressure had a decelerating effect. One possible explanation is that the adsorption of butanol at low pressure is relatively small and increases with pressure. At high pressures the surface of the catalyst becomes virtually covered; further increases in pressure would exert relatively little effect on the surface concentration of the reacting molecules provided only the adsorption step were rate-controlling. In this event the rate of decomposition would become virtually constant (5). However, as the pressure is increased, the rate of surface reaction and possibly the rate of desorption of products (particularly water) may begin to have a pronounced effect on the over-all reaction rate and may possibly account for the decreased decompositions at pressures of 2500 lb./sq.in. and greater. It is improbable that diffusion affects the over-all reaction rate at temperatures as high as 750° F. Furthermore diffusion proc-

esses have much lower activation energies (6) than the probable value of 36,500 cal. for the decomposition of alcohol (1).

Because of the lack of sufficient data, particularly in the regions of low conversions, no attempts were made to obtain a more complete, quantitative analysis of the kinetics of the over-all decomposition reaction.

Conclusions

1. Continuous operation of this small-scale plant at elevated pressures (10,000 lb./sq.in.) and temperatures (1200° F.) confirmed the applicability of this equipment for studying chemical reactions over a wide range of pressures, temperatures, and feed rates.

2. For a given feed rate the decomposition of butanol-1 over an alumina-silicate cracking catalyst at 760° F. increases with increasing pressure and thereafter decreases with increasing pressures. Maximum decomposition occurs at some pressure between 750 and 2500 lb./sq.in.

3. The major reaction in the decomposition of butanol-1, over the range of conditions investigated, is the direct dehydration to mixed butenes and water. The butenes are entirely responsible for any side reactions that occur; these reactions probably involve the condensation of butenes to form higher molecular weight substances. Cracking of the butenes to lighter substances proceeds only to a limited extent.

4. The extent of the side reactions increases with increasing pressures and decreasing feed rates at 760° F.

5. For pressures up to 750 lb./sq.in. gage the adsorption of butanol may be the rate-controlling step. For increasing pressures above 2500 lb./sq.in. gage the rate of surface reaction or the desorption of the water appears to affect the adsorption step to such an extent that the over-all rate of decomposition progressively decreases with pressure.

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SYNTHESIS OF METHANE FROM CARBON DIOXIDE AND HYDROGEN

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Rate of formation of methane from carbon dioxide and hydrogen was measured by passing feed gas ranging in composition from 80% H₂ and 20% CO₂ to 30% H₂ and 70% CO₂ over a reduced nickel catalyst in a continuous apparatus. Several runs were made in which the feed gas contained methane. The space time was varied from 0 to 8×10^{-5} hr. Temperature range investigated was 500-750° F., and the pressure was maintained at one atmosphere in all runs. Average rates of methane formation obtained by differentiation of the data vary from 0 to 5.60 lb. moles/(day) (lb. catalyst).

Rate of reaction can be correlated by a mechanism which assumes that the major resistance to the over-all process is offered by the reaction of an adsorbed carbon dioxide molecule with at least two adsorbed hydrogen molecules.

The following equations for the rate of methane formation result from the extremes of the range of correlating mechanisms:

$$r = \frac{C_1 \left(p_{\text{CO}_2} p_{\text{H}_2}^2 - \frac{p_{\text{CH}_4} p_{\text{H}_2}^2}{K_1} \right)}{(p_{\text{H}_2}^{1/2} + C_2 p_{\text{CO}_2} + C_3)^2}$$

$$r = \frac{C_1' \left(p_{\text{CO}_2} p_{\text{H}_2}^4 - \frac{p_{\text{CH}_4} p_{\text{H}_2}^2}{K_1} \right)}{(p_{\text{H}_2}^{1/2} + C_2' p_{\text{CO}_2} + C_3')^2}$$

THE ever-increasing demand for natural gas, both as a fuel and as a chemical raw material foreshadows increasing costs and the eventual exhaustion of gas reserves. When the conversion of coal to gaseous fuels becomes economically attractive, it appears likely that the catalytic upgrading of the mixtures of carbon monoxide, carbon dioxide and hydrogen will be advisable. Reactions of carbon monoxide and carbon dioxide with hydrogen to form methane are of major interest in this enrichment.

The kinetics of the reaction between carbon monoxide and hydrogen has been investigated previously by Akers and White (1). Previous investigations of the reaction between carbon dioxide and hydrogen (3,4, 8-13) are primarily

qualitative in nature and present no data suitable for a kinetic analysis. The purpose of this investigation was to obtain a large amount of precise data over a wide range of temperature, composition and space velocity in order to establish the kinetics of the reaction between carbon dioxide and hydrogen over the same reduced nickel catalyst previously used (1) in the investigation of the reaction between carbon monoxide and hydrogen.

Experimental Apparatus. A flow diagram of the flow system used in this investigation is shown in Figure 1.

Two commercial gas cylinders supply each of two feed lines. Gas flows through two pressure-reducing valves in series in each of these lines, through an orifice, through a control valve and through a flow controller.

After leaving the flow controllers, the gases mix and flow to the preheater. Upon leaving the preheater the feed gas enters the reactor and flows downward through the catalyst bed.

The reactor consists of a 30-in. length of standard 3/4-in. seamless steel pipe fitted at

each end with a steel union closure. Feed gas enters through a steel nipple welded into the side of the reactor at its upper end. Product gas leaves through a similar connection at the lower end. A 24-in. length of standard 4-in. seamless steel pipe filled with Dowtherm jackets the central portion of the reactor. A 2500 watt-230v. G.E. Calrod heater has been coiled and mounted inside the jacket.

A small water-cooled condenser is attached to the side of the jacket at its upper end to condense the Dowtherm vapor generated within the jacket. The upper end of this condenser also serves as the inlet for sufficient gas to pressurize the Dowtherm so that it boils at a desired control temperature.

A perforated disc supports the catalyst within the reactor. This disc is attached by means of a set screw to a 3/4-in. O.D. \times .028-in. wall steel tube which runs the length of the reactor and serves as a well for a traveling 36-in. Leeds and Northrup miniature pipe-type thermocouple.

After leaving the reactor the product gas flows through a water-cooled condenser with a condensate trap attached to the bottom, bubbles through water to assure saturation, is metered in a calibrated Sargent wet test meter filled with water, and vents to the flue.

Gas samples can be withdrawn directly into an Orsat apparatus from a tap located just downstream from the wet test meter. A by-pass around the reactor enables the metering and analysis of the feed gas to take place through the same meter and sampling line.

Fixed iron-constantan thermocouples indicate the temperature of the boiling Dowtherm, the temperature of the preheater, and the temperature of the exit gas from the humidifier. The traveling thermocouple is of iron constantan.

Materials. The hydrogen, carbon dioxide and methane used are obtained from commercial cylinders. The hydrogen, produced by Linde Air Products Co., contains 0.1% O₂ as its only impurity. The carbon dioxide, manufactured by Pure Carbonic, Inc., is 99.5% pure. The methane, obtained from the Matheson Co., Inc., contains 4% of a mixture of ethane, nitrogen and carbon dioxide.

The reduced nickel catalyst, Harshaw Ni-88 Tablets 3/4-in., was donated by the Harshaw Chemical Co. and was taken from the same lot as that used in the synthesis of methane from carbon monoxide and hydrogen (1). The 3/4-in. cylindrical pellets have an average weight of .0475 g. and a bulk density of 1.511 g./cc.

Note: Complete data on Tables 1, 2, 3, and 5 are on file Document (2824) with the American Documentation Institute, 1719 N Street, Northwest, Washington, D. C. Data can be obtained by remitting 50 cents for microfilm and \$1.00 for photoprint.

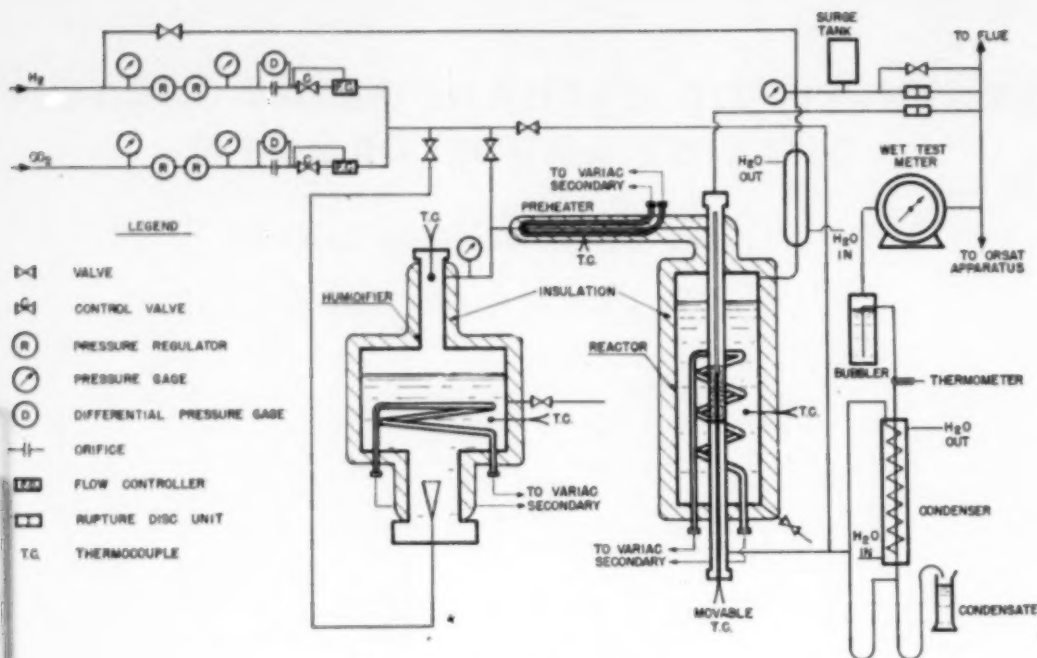


Fig. 1. Flow sheet of reaction equipment.

Experimental Procedure. A predetermined amount of fresh catalyst is thoroughly mixed with sufficient copper granules to bring the total volume to 50 cc. and charged to the reactor. The Dowtherm heater is energized and the feed rate and composition are adjusted to the desired values while the reactor is coming up to temperature.

When the feed is properly regulated and the Dowtherm is boiling steadily, usually about 497° F., the feed is admitted to the reactor and the by-pass is closed. Reaction is noticed immediately by a decrease in metered volume. Steady-state conditions are indicated by the approach of the product

rate to a constant value, usually somewhat above that noted immediately after flow is admitted to the reactor. The following data are obtained when these conditions are reached.

1. Regulated pressures of hydrogen and carbon dioxide feed streams
2. Time per revolution of the wet test meter
3. Mole per cent carbon dioxide (dry basis) in product gas
4. Temperature of boiling Dowtherm
5. Temperature of gas being metered
6. Barometric pressure

7. Temperature distribution throughout the catalyst bed

After the above data are recorded, the reactor jacket is closed from the atmosphere, pressurized, and brought to a new boiling point at 550° F. The previously described procedure is repeated and the reactor-jacket temperature increased in increments of 50° F.

After a run is completed at 750° F., the feed is by-passed around the reactor in order to check its flow rate and composition. When the reactor has cooled, the jacket pressure is bled to the atmosphere, and the catalyst is removed in preparation for the next series of runs.

TABLE 1.—EXPERIMENTAL RESULTS

Feed Composition: 20% CO₂-80% H₂

| Run No. (1) | Feed Composition | | | | Catalyst Number of Pellets (6) | Jacket Temp. ° F. (7) | Product Rate std. cu. ft. dry gas/hr (8) | CO ₂ in Product (Dry Basis) mole % (9) | Product Gas Composition (Moles %) | | | | | |
|-------------|----------------------------|---------------------------|----------------------------|--------|--------------------------------|-----------------------|--|---|-----------------------------------|---------|---------------------|----------------------|-----------------------|-------------|
| | CO ₂ mole % (2) | H ₂ mole % (3) | CH ₄ mole % (4) | | | | | | CO ₂ (10) | CO (11) | H ₂ (12) | CH ₄ (13) | H ₂ O (14) | Inerts (15) |
| 3 | 22.0 | 80.0 | | 135.5 | 0 | 551 | 135.2 | 10.25 | 10.05 | 0.05 | 78.6 | 0.15 | 1.25 | .. |
| 8 | 20.1 | 79.9 | | 134.9 | 85 | 546 | 127.5 | 19.7 | 19.15 | 0.1 | 76.4 | 1.4 | 2.95 | .. |
| 14 | 20.1 | 79.9 | | 138.25 | 75 | 551 | 118.3 | 19.3 | 17.9 | 0.18 | 71.35 | 8.5 | 7.1 | .. |
| 20 | 20.0 | 80.0 | | 135.8 | 150 | 551 | 110.1 | 18.8 | 16.85 | 0.1 | 67.35 | 5.3 | 10.5 | .. |
| 26 | 20.0 | 80.0 | | 134.75 | 360 | 551 | 94.3 | 18.0 | 14.8 | 0.0 | 58.75 | 8.85 | 17.6 | .. |
| 29 | 20.0 | 80.0 | | 136.1 | 400 | 550 | 78.8 | 16.35 | 12.0 | 0.0 | 48.05 | 13.35 | 26.9 | .. |
| 4 | 20.0 | 80.0 | | 134.8 | 0 | 650 | 132.2 | 18.6 | 18.25 | 1.75 | 78.1 | 0.05 | 1.85 | .. |
| 10 | 20.1 | 79.9 | | 134.4 | 25 | 648 | 116.0 | 18.7 | 17.35 | 0.75 | 70.85 | 5.45 | 7.7 | .. |
| 16 | 20.2 | 79.8 | | 135.15 | 75 | 650 | 98.45 | 17.7 | 14.9 | 0.8 | 60.6 | 7.65 | 16.05 | .. |
| 22 | 20.0 | 80.0 | | 135.2 | 150 | 650 | 87.7 | 17.35 | 13.65 | 0.1 | 54.25 | 10.65 | 21.35 | .. |
| 28 | 20.1 | 79.9 | | 134.85 | 250 | 650 | 68.6 | 15.2 | 10.25 | 0.05 | 40.85 | 16.25 | 32.6 | .. |
| 34 | 20.0 | 80.0 | | 134.65 | 400 | 650 | 49.85 | 11.55 | 6.1 | 0.05 | 24.8 | 23.0 | 46.05 | .. |

Feed Composition: 20.5% CO₂-66.5% H₂-13% CH₄

| | | | | | | | | | | | | | | |
|-----|------|------|------|--------|-----|-----|-------|-------|-------|------|-------|-------|-------|----|
| 130 | 20.6 | 66.4 | 13.0 | 139.15 | 25 | 652 | 117.9 | 10.65 | 18.75 | 0.0 | 55.3 | 16.25 | 9.3 | .. |
| 136 | 20.6 | 66.4 | 13.0 | 137.55 | 75 | 650 | 98.0 | 14.3 | 15.2 | 0.5 | 43.9 | 23.4 | 17.0 | .. |
| 142 | 21.0 | 65.7 | 13.3 | 138.00 | 150 | 648 | 79.4 | 17.20 | 12.20 | 0.85 | 29.40 | 29.95 | 27.30 | .. |

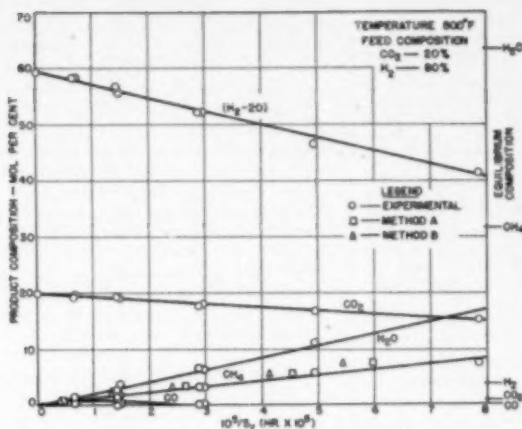


Fig. 2. Product composition vs. space time for a 20% CO_2 -80% H_2 at 1 atm. and 500° F.

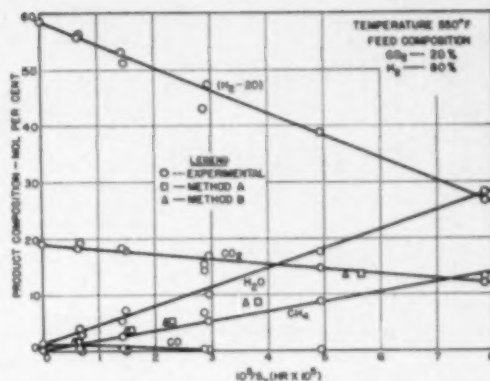


Fig. 3. Product composition vs. space time for a 20% CO_2 -80% H_2 at 1 atm. and 550° F.

Methods of Analysis

A feed analysis was made before each series of six runs. This analysis, with minor corrections for unavoidable fluctuations in the regulated pressures of the independent feed streams, applied to all six runs. In analyzing the feed, carbon dioxide was determined by absorption in potassium hydroxide solution in a conventional Orsat apparatus. Hydrogen was obtained by difference taking into account impurities in the commercial gas used as the source of feed. The purity claimed for each gas was confirmed by Orsat and mass spectrographic analyses.

The carbon dioxide in the product gas was obtained also in the Orsat apparatus but all other components were determined by material balances around the reactor. The carbon formed in the reactor was negligible and mass spectrographic analysis assured that no hydrocarbons other than methane were formed. The system was thereby restricted to a maximum of five components; namely, carbon dioxide, carbon monoxide, hydrogen, methane, and water. Carbon, oxygen, and hydrogen balances permit the amount of any product to be expressed as a function of the amounts

of carbon dioxide and hydrogen that have reacted.

Experimental Results

Table I presents typical experimental data of this investigation. The run numbers are listed in Column 1. Each run is obtained under steady-state conditions and consists of an analysis of the feed and product gases at a given feed rate, temperature, pressure and quantity of catalyst. Columns 2-4 present the feed gas compositions. Four different feed mixtures were used, one mixture containing the product methane which was introduced in constant proportion to the carbon dioxide. Column 5 presents the rates of feed which are approximately the same for all runs. Column 6 gives the quantities of catalyst used in each run. Column 7 lists the jacket temperatures. Column 8 presents the rates of product (dry basis). Column 9 lists the mole per cents of carbon dioxide in the product gas (dry basis). The complete product gas analyses are tabulated (on a mole per cent basis) in Columns 10-15. The plots of gas composition versus space time are shown in Figures 2-25.

No definite decrease in catalyst activity

with time was observed during any series of runs. A seven-day test at approximately 600° F. of the activity of this catalyst when used to synthesize methane from carbon monoxide and hydrogen was made and reported previously (1).

Calculated Results

Table 2 presents typical average rates of formation of each component as determined by differentiation of the experimental data. Runs (Column 1) are listed in order of increasing space time, (Column 3) in groups of constant temperature, (Column 2) and constant feed composition.

Ratios of the increments of carbon dioxide and hydrogen conversion (Columns 7 and 10) to the corresponding increments of catalyst (Column 5) are average rates of reaction over each increment for these components and are listed in Columns 8 and 11. Columns 12 and 13 present these rates in more conventional units. Average rates of methane, water and carbon monoxide formation (Columns 14-16) are obtained by a material balance.

A typical plot of the average rate of methane formation as a function of space

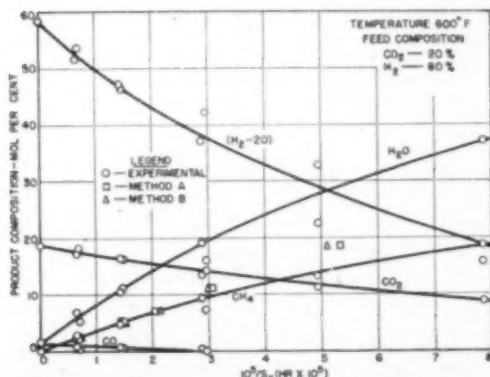


Fig. 4. Product composition vs. space time for a 20% CO_2 -80% H_2 feed at 1 atm. and 600° F.

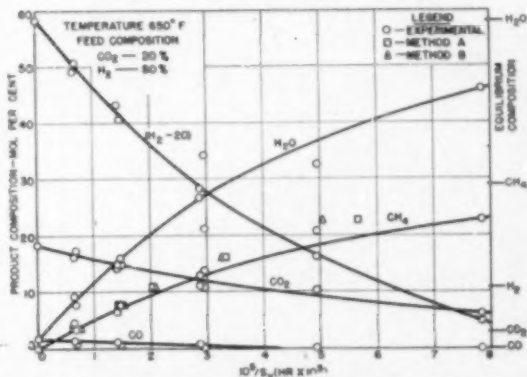


Fig. 5. Product composition vs. space time for a 20% CO_2 -80% H_2 feed at 1 atm. and 650° F.

TABLE 3.—SUMMARY OF RATE EQUATION CONSTANTS

| Feed Composition: 20% CO, 80% H ₂ | | | | | | | | | |
|--|----------------|---------------------------------------|---------------------------|----|----------------------------------|------------------|---------------------------|---------------------------------|---|
| Average Temp. (°F.) | Reactor Number | Space Time (hr) (20% H ₂) | Catalyst Number of Pounds | ΔS | CO ₂ Reacted (lb/day) | ΔCO ₂ | 100 ΔCO ₂ / ΔS | H ₂ Reacted (lb/day) | Average Rate of Formation (lb/day) (100% H ₂) |
| 500 | 1 | 0.000 | 0 | 35 | 1.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 2 | 0.000 | 0 | 35 | 2.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 3 | 0.000 | 0 | 40 | 2.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 4 | 0.000 | 0 | 40 | 3.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 5 | 0.000 | 0 | 40 | 3.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 6 | 0.000 | 0 | 40 | 4.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 7 | 0.000 | 0 | 40 | 4.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 8 | 0.000 | 0 | 40 | 5.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 9 | 0.000 | 0 | 40 | 5.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 10 | 0.000 | 0 | 40 | 6.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 11 | 0.000 | 0 | 40 | 6.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 12 | 0.000 | 0 | 40 | 7.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 13 | 0.000 | 0 | 40 | 7.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 14 | 0.000 | 0 | 40 | 8.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 15 | 0.000 | 0 | 40 | 8.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 16 | 0.000 | 0 | 40 | 9.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 17 | 0.000 | 0 | 40 | 9.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 18 | 0.000 | 0 | 40 | 10.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 19 | 0.000 | 0 | 40 | 10.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 20 | 0.000 | 0 | 40 | 11.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 21 | 0.000 | 0 | 40 | 11.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 22 | 0.000 | 0 | 40 | 12.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 23 | 0.000 | 0 | 40 | 12.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 24 | 0.000 | 0 | 40 | 13.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 25 | 0.000 | 0 | 40 | 13.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 26 | 0.000 | 0 | 40 | 14.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 27 | 0.000 | 0 | 40 | 14.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 28 | 0.000 | 0 | 40 | 15.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 29 | 0.000 | 0 | 40 | 15.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 30 | 0.000 | 0 | 40 | 16.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 31 | 0.000 | 0 | 40 | 16.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 32 | 0.000 | 0 | 40 | 17.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 33 | 0.000 | 0 | 40 | 17.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 34 | 0.000 | 0 | 40 | 18.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 35 | 0.000 | 0 | 40 | 18.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 36 | 0.000 | 0 | 40 | 19.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 37 | 0.000 | 0 | 40 | 19.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 38 | 0.000 | 0 | 40 | 20.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 39 | 0.000 | 0 | 40 | 20.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 40 | 0.000 | 0 | 40 | 21.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 41 | 0.000 | 0 | 40 | 21.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 42 | 0.000 | 0 | 40 | 22.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 43 | 0.000 | 0 | 40 | 22.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 44 | 0.000 | 0 | 40 | 23.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 45 | 0.000 | 0 | 40 | 23.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 46 | 0.000 | 0 | 40 | 24.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 47 | 0.000 | 0 | 40 | 24.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 48 | 0.000 | 0 | 40 | 25.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 49 | 0.000 | 0 | 40 | 25.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 50 | 0.000 | 0 | 40 | 26.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 51 | 0.000 | 0 | 40 | 26.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 52 | 0.000 | 0 | 40 | 27.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 53 | 0.000 | 0 | 40 | 27.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 54 | 0.000 | 0 | 40 | 28.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 55 | 0.000 | 0 | 40 | 28.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 56 | 0.000 | 0 | 40 | 29.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 57 | 0.000 | 0 | 40 | 29.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 58 | 0.000 | 0 | 40 | 30.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 59 | 0.000 | 0 | 40 | 30.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 60 | 0.000 | 0 | 40 | 31.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 61 | 0.000 | 0 | 40 | 31.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 62 | 0.000 | 0 | 40 | 32.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 63 | 0.000 | 0 | 40 | 32.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 64 | 0.000 | 0 | 40 | 33.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 65 | 0.000 | 0 | 40 | 33.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 66 | 0.000 | 0 | 40 | 34.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 67 | 0.000 | 0 | 40 | 34.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 68 | 0.000 | 0 | 40 | 35.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 69 | 0.000 | 0 | 40 | 35.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 70 | 0.000 | 0 | 40 | 36.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 71 | 0.000 | 0 | 40 | 36.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 72 | 0.000 | 0 | 40 | 37.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 73 | 0.000 | 0 | 40 | 37.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 74 | 0.000 | 0 | 40 | 38.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 75 | 0.000 | 0 | 40 | 38.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 76 | 0.000 | 0 | 40 | 39.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 77 | 0.000 | 0 | 40 | 39.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 78 | 0.000 | 0 | 40 | 40.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 79 | 0.000 | 0 | 40 | 40.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 80 | 0.000 | 0 | 40 | 41.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 81 | 0.000 | 0 | 40 | 41.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 82 | 0.000 | 0 | 40 | 42.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 83 | 0.000 | 0 | 40 | 42.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 84 | 0.000 | 0 | 40 | 43.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 85 | 0.000 | 0 | 40 | 43.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 86 | 0.000 | 0 | 40 | 44.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 87 | 0.000 | 0 | 40 | 44.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 88 | 0.000 | 0 | 40 | 45.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 89 | 0.000 | 0 | 40 | 45.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 90 | 0.000 | 0 | 40 | 46.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 91 | 0.000 | 0 | 40 | 46.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 92 | 0.000 | 0 | 40 | 47.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 93 | 0.000 | 0 | 40 | 47.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 94 | 0.000 | 0 | 40 | 48.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 95 | 0.000 | 0 | 40 | 48.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 96 | 0.000 | 0 | 40 | 49.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 97 | 0.000 | 0 | 40 | 49.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 98 | 0.000 | 0 | 40 | 50.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 99 | 0.000 | 0 | 40 | 50.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 100 | 0.000 | 0 | 40 | 51.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 101 | 0.000 | 0 | 40 | 51.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 102 | 0.000 | 0 | 40 | 52.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 103 | 0.000 | 0 | 40 | 52.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 104 | 0.000 | 0 | 40 | 53.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 105 | 0.000 | 0 | 40 | 53.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 106 | 0.000 | 0 | 40 | 54.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 107 | 0.000 | 0 | 40 | 54.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 108 | 0.000 | 0 | 40 | 55.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 109 | 0.000 | 0 | 40 | 55.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 110 | 0.000 | 0 | 40 | 56.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 111 | 0.000 | 0 | 40 | 56.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 112 | 0.000 | 0 | 40 | 57.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 113 | 0.000 | 0 | 40 | 57.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 114 | 0.000 | 0 | 40 | 58.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 115 | 0.000 | 0 | 40 | 58.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 116 | 0.000 | 0 | 40 | 59.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 117 | 0.000 | 0 | 40 | 59.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 118 | 0.000 | 0 | 40 | 60.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 119 | 0.000 | 0 | 40 | 60.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 120 | 0.000 | 0 | 40 | 61.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 121 | 0.000 | 0 | 40 | 61.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 122 | 0.000 | 0 | 40 | 62.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 123 | 0.000 | 0 | 40 | 62.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 124 | 0.000 | 0 | 40 | 63.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 125 | 0.000 | 0 | 40 | 63.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 126 | 0.000 | 0 | 40 | 64.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 127 | 0.000 | 0 | 40 | 64.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 128 | 0.000 | 0 | 40 | 65.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 129 | 0.000 | 0 | 40 | 65.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 130 | 0.000 | 0 | 40 | 66.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 131 | 0.000 | 0 | 40 | 66.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 132 | 0.000 | 0 | 40 | 67.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 133 | 0.000 | 0 | 40 | 67.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 134 | 0.000 | 0 | 40 | 68.0 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500 | 135 | 0.000 | 0 | 40 | 68.5 | 0.5 | 1.450 | 2.1 | 1.365 |
| 500</ | | | | | | | | | |

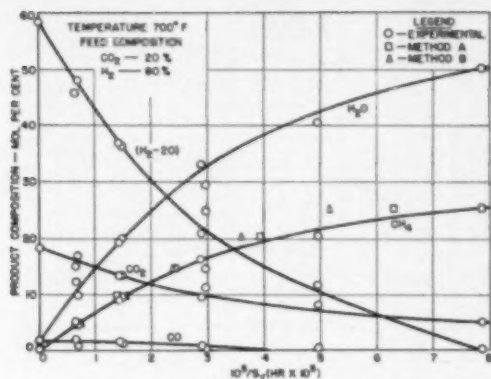


Fig. 6. Product composition vs. space time for a 20% CO₂-80% H₂ feed at 1 atm. and 700° F.

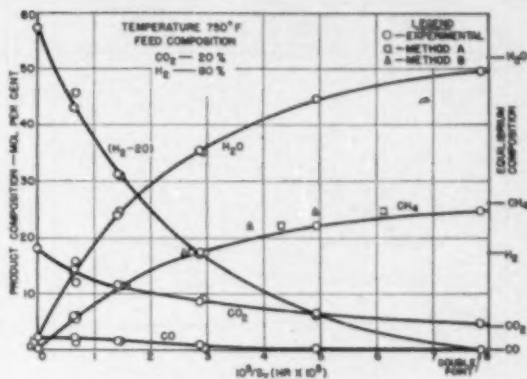


Fig. 7. Product composition vs. space time for a 20% CO₂-80% H₂ feed at 1 atm. and 750° F.

$$r = \frac{K_{H_2} L \left[p_{H_2} - \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/2} \right]}{4 \left[1 + K_{H_2} \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/2} + K_{CO_2} p_{CO_2} + K_{CH_4} + K_{H_2O} p_{H_2O} \right]} \quad (2)$$

$$r = \frac{k_{H_2} s L \left[p_{H_2} - \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/2} \right]}{8 \left[1 + K_{H_2} \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/2} + K_{CO_2} p_{CO_2} + K_{CH_4} p_{CH_4} + K_{H_2O} p_{H_2O} \right]} \quad (3)$$

$$r = \frac{k_{H_2} s L \left[p_{H_2}^{1/2} - \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/4} \right]}{8 \left[1 + K_{H_2}^{1/2} \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/4} + K_{CO_2} p_{CO_2} + K_{CH_4} p_{CH_4} + K_{H_2O} p_{H_2O} \right]} \quad (4)$$

$$r = \frac{k_{H_2} s L \left[p_{H_2} - \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/2} \right]}{8 \left[1 + K_{H_2}^{1/2} \left(\frac{p_{CH_4} p_{H_2O}^2}{K_1 p_{CO_2}} \right)^{1/4} + K_{CO_2} p_{CO_2} + K_{CH_4} p_{CH_4} + K_{H_2O} p_{H_2O} \right]} \quad (5)$$

Considering mechanism 1, Equation (2), for all feed mixtures containing only carbon dioxide and hydrogen, the initial partial pressures of methane and water were zero and the initial partial pressure of carbon dioxide was equal to the total pressure minus the partial pressure of hydrogen. Under these conditions Equation (2) reduces to

$$r^0 = \left(\frac{k_{H_2} L}{4} \right) \frac{p_{H_2}^0}{(1 + PK_{CO_2}) - K_{CO_2} p_{H_2}^0} \quad (6)$$

where

r^0 = initial rate of methane formation
 $p_{H_2}^0$ = initial partial pressure of hydrogen

If Equation (6) were applicable, an increase in the partial pressure of hydrogen in the feed would result in a corresponding increase in the initial rate of methane formation. Rates of methane formation calculated from the experimental data, however, indicate that the

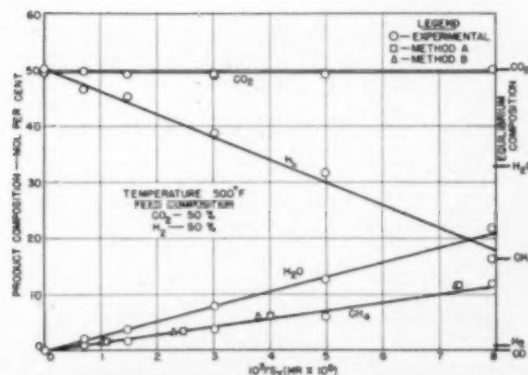


Fig. 8. Product composition vs. space time for a 50% CO₂-50% H₂ feed at 1 atm. and 500° F.

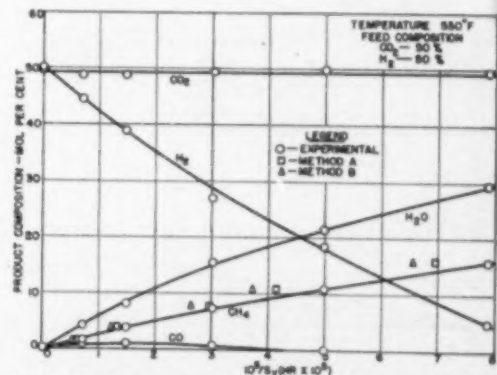


Fig. 9. Product composition vs. space time for a 50% CO₂-50% H₂ feed at 1 atm. and 550° F.

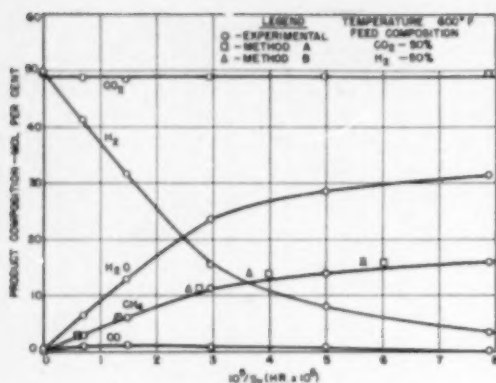


Fig. 10. Product composition vs. space time for a 50% CO_2 -50% H_2 feed at 1 atm. and 600° F.

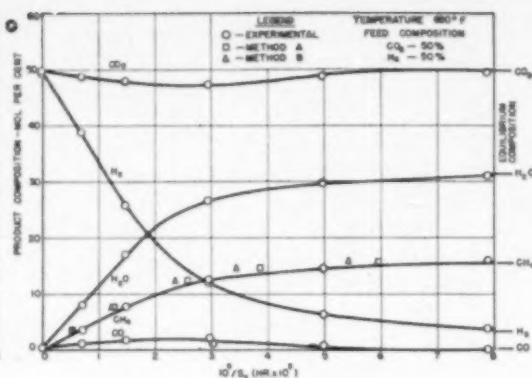


Fig. 11. Product composition vs. space time for a 50% CO_2 -50% H_2 feed at 1 atm. and 650° F.

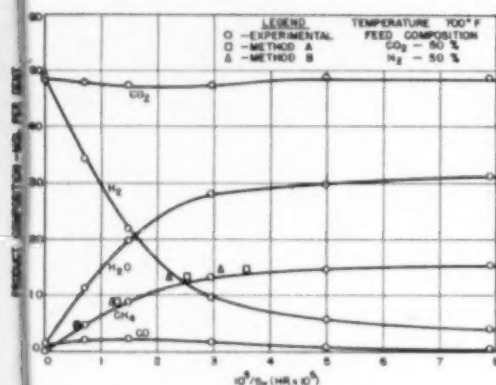


Fig. 12. Product composition vs. space time for a 50% CO_2 -50% H_2 feed at 1 atm. and 700° F.

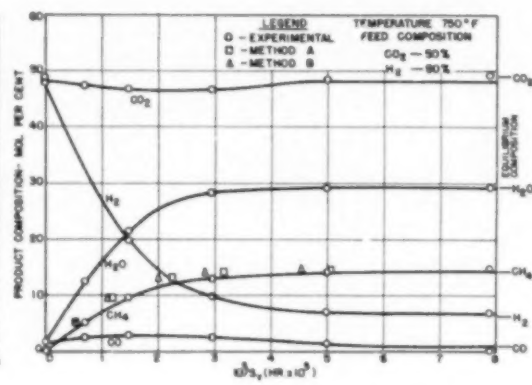


Fig. 13. Product composition vs. space time for a 50% CO_2 -50% H_2 feed at 1 atm. and 750° F.

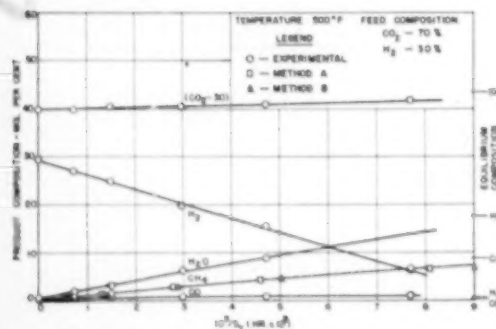


Fig. 14. Product composition vs. space time for a 70% CO_2 -30% H_2 feed at 1 atm. and 500° F.

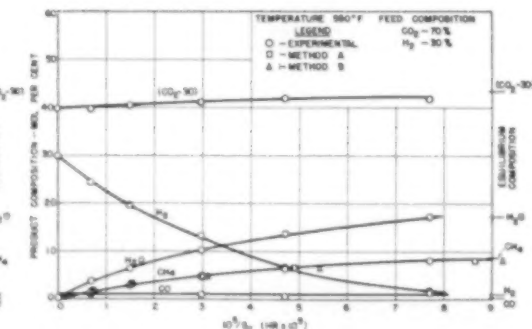


Fig. 15. Product composition vs. space time for a 70% CO_2 -30% H_2 feed at 1 atm. and 550° F.

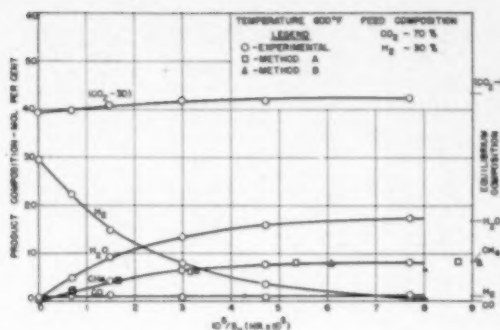


Fig. 16. Product composition vs. space time for a 70% CO -30% H_2 feed at 1 atm. and 600° F.

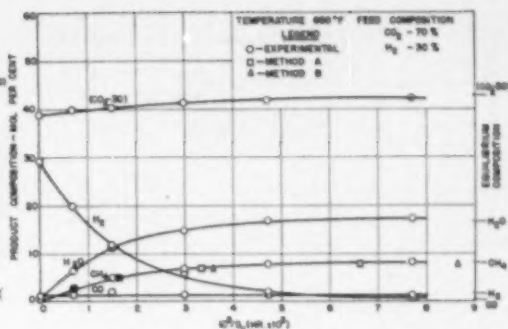


Fig. 17. Product composition vs. space time for a 70% CO -30% H_2 feed at 1 atm. and 650° F.

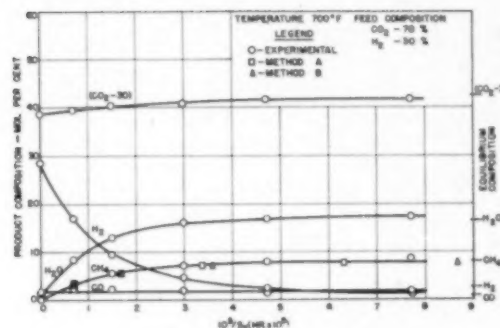


Fig. 18. Product composition vs. space time for a 70% CO -30% H_2 feed at 1 atm. and 700° F.

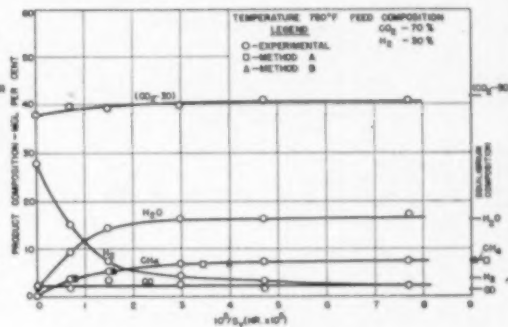


Fig. 19. Product composition vs. space time for a 70% CO -30% H_2 feed at 1 atm. and 750° F.

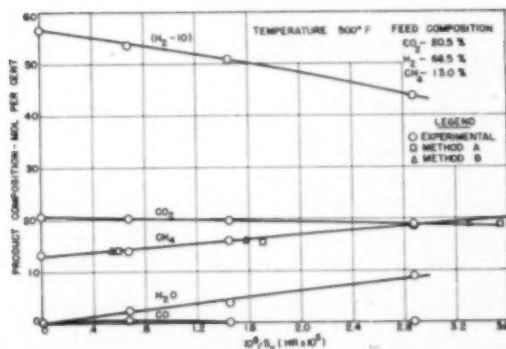


Fig. 20. Product composition vs. space time for a 20.5% CO -66.5% H_2 -13% CH_4 feed at 1 atm. and 500° F.

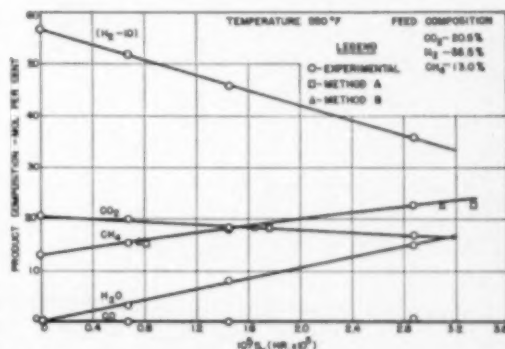


Fig. 21. Product composition vs. space time for a 20.5% CO -66.5% H_2 -13% CH_4 feed at 1 atm. and 550° F.

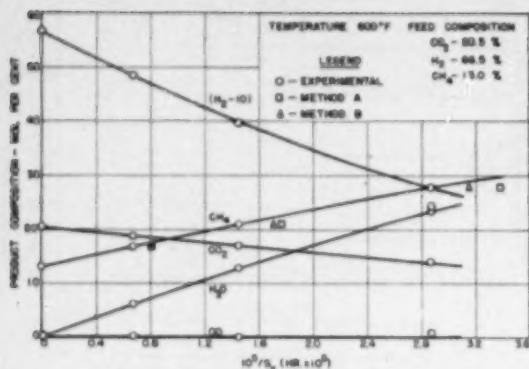


Fig. 22. Product composition vs. space time for a 20.5% CO_2 -66.5% H_2 -13% CH_4 feed at 1 atm. and 600° F.

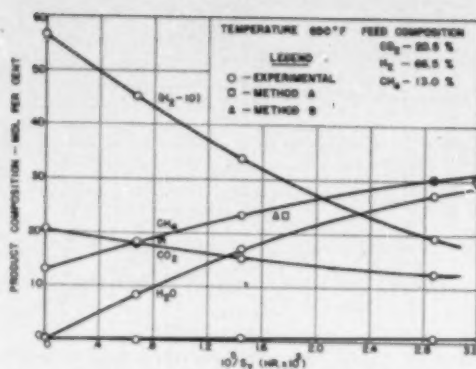


Fig. 23. Product composition vs. space time for a 20.5% CO_2 -66.5% H_2 -13% CH_4 feed at 1 atm. and 650° F.

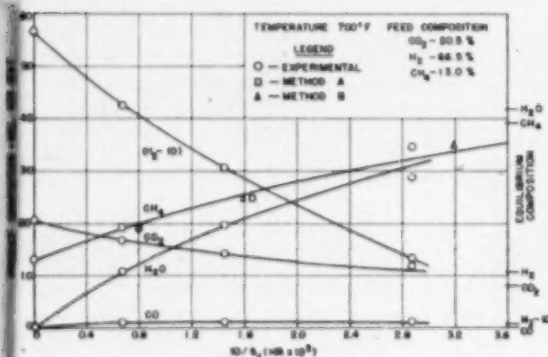


Fig. 24. Product composition vs. space time for a 20.5% CO_2 -66.5% H_2 -13% CH_4 feed at 1 atm. and 700° F.

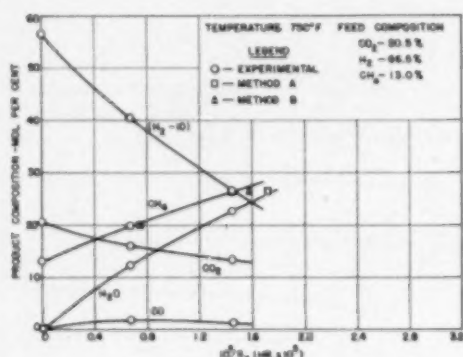


Fig. 25. Product composition vs. space time for a 20.5% CO_2 -66.5% H_2 -13% CH_4 feed at 1 atm. and 750° F.

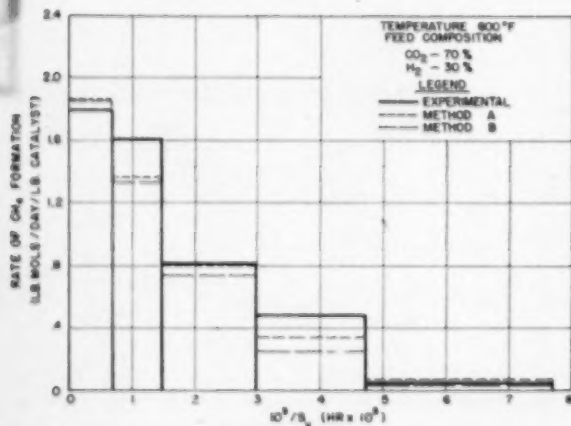


Fig. 26. Average rate of methane formation vs. space time for a 70%-30% H_2 feed at 1 atm. and 600° F.

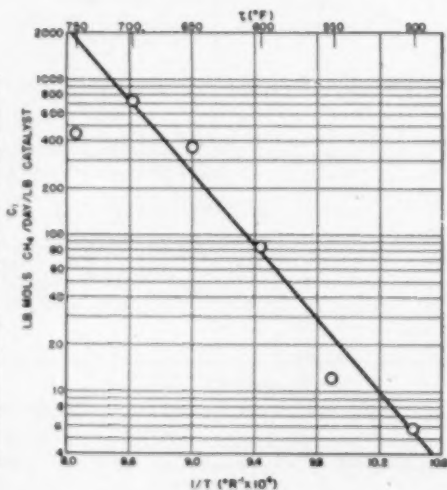


Fig. 27. Variation of rate-equation constant C_1 with temperature.

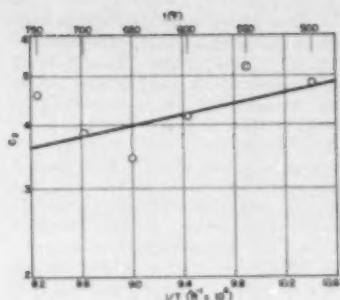


Fig. 28. Variation of rate-equation constant C_s with temperature.

initial rate passes through a maximum as the hydrogen concentration in the feed is increased. Equations (3), (4) and (5), although differing somewhat in form, all lead to the conclusion that if any of them were valid, the initial rate would increase continuously with increasing hydrogen in the feed. An equation similar to Equation (6) can be derived for the case of carbon dioxide adsorption as the rate-controlling step which is invalid for the same reasons presented for Equation (6), and it is therefore concluded that the adsorption of a reactant is not rate-controlling.

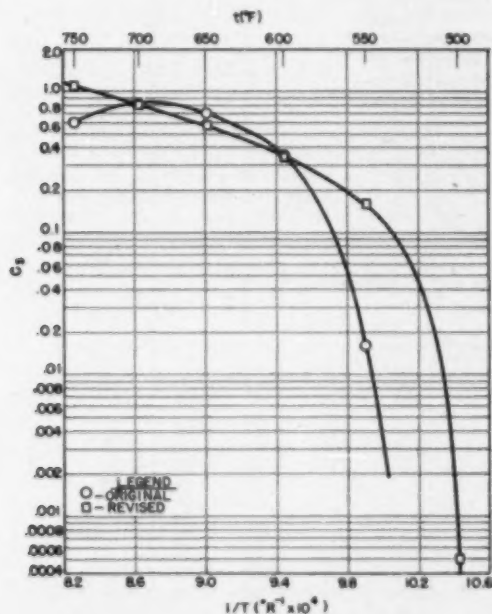


Fig. 29. Variation of rate-equation constant C_s with temperature.

Desorption of Products. If the desorption of methane were rate-controlling, the rate of reaction would be given by

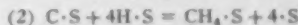
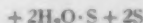
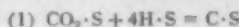
$$r = \frac{k_{CH_4} L \left(\frac{K_1 p_{CO_2} p_{H_2}^4}{p_{H_2O}^5} - p_{CH_4} \right)}{1 + K_{H_2} p_{H_2} + K_{CO_2} p_{CO_2} + K_{CH_4} K_1 \frac{p_{CO_2} p_{H_2}^4}{p_{H_2O}^5} + K_{H_2O} p_{H_2O}} \quad (7)$$

The method of least squares was used to evaluate the constants in Equation (7) from the data at 650° F.

Some of the constants were negative and since by definition these constants must be positive, the desorption of methane cannot be considered as the rate-controlling step. The desorption of water was rejected as a possible rate-controlling step after the above procedure produced a similar result.

Surface Reaction. Surface reaction often offers the major resistance to the over-all process. The form of the de-

rived rate equation depends upon the mechanism assumed for the surface reaction.



with step (1) rate-controlling and with equilibrium maintained in step (2), the following equation may be derived where step (1) represents the reaction between an adsorbed carbon dioxide

TABLE 4.—THERMODYNAMIC PROPERTIES

| Method | Std. Enthalpy Change of Adsorption B.t.u./lb.mole | | Std. Entropy Change of Adsorption B.t.u./ (lb.mole) (° F.) | | Std. Enthalpy of Activation B.t.u./lb.mole ΔH^\ddagger |
|--------|---|--------|--|-------|--|
| | (1) | (2) | (3) | (4) | |
| A | 24,500 | 19,700 | 35.5 | 20.8 | 50,000 |
| B | 24,250 | 16,200 | 15.8 | 14.11 | 33,350 |

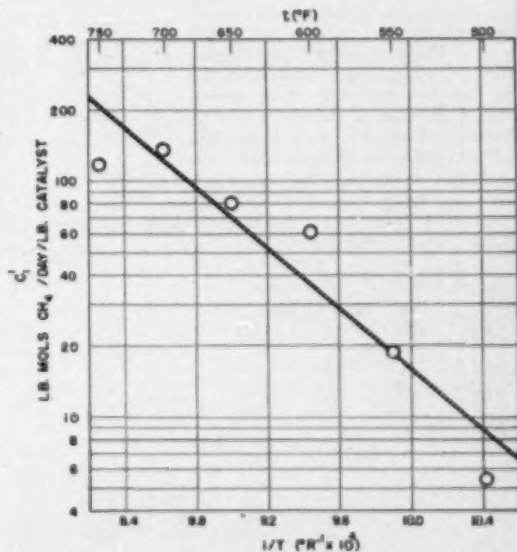


Fig. 30. Variation of rate-equation constant C_s' with temperature.

molecule and two dissociated hydrogen molecules

$$r = \frac{k_1(s-1)(s-2)(s-3)K_{CO_2}K_{H_2}^2}{(1 + K_{H_2}p_{H_2} + K_{CO_2}p_{CO_2} + K_{CH_4}p_{CH_4} + K_{H_2O}p_{H_2O})^5} p_{CO_2}p_{H_2}^2 - \frac{p_{CH_4}p_{H_2O}^2}{K_1p_{H_2}^2} \quad (8)$$

The rates of methane formation over the first space-time increment for the feed mixture containing methane did not differ appreciably from those for a feed mixture containing the same concentration of carbon dioxide, no methane, and a considerably higher percentage of hydrogen. Equation (8) could be valid under these conditions only if the adsorption equilibrium constant, K_{CH_4} , were negligible compared with the adsorption equilibrium constant, K_{H_2O} . A run was also made with a feed containing 50% H_2O that indicated that K_{H_2O} must also be negligible compared to K_{H_2} . Neglecting the terms in the denominator involving K_{CH_4} and K_{H_2O} , Equation (8) becomes

$$r = \frac{C(p_{CO_2}p_{H_2}^2 - p_{CH_4}p_{H_2O}^2/K_1p_{H_2}^2)}{(1 + \sqrt{K_{H_2}p_{H_2} + K_{CO_2}p_{CO_2}})^5} \quad (9)$$

which can be written in the following linear form:

$$r = \frac{C_1^{1/5}p_{H_2}^{4/5}}{p_{H_2}^{1/5}} (p_{CO_2}p_{H_2}^2 - p_{CH_4}p_{H_2O}^2/K_1p_{H_2}^2)^{1/5} - C_2r^{1/5}p_{CO_2}^{1/5} - C_3r^{1/5}p_{H_2}^{1/5} \quad (10)$$

where

$$C_1 = C/K_{H_2}^{4/5} \\ C_2 = K_{CO_2}/K_{H_2}^{1/5} \\ C_3 = 1/K_{H_2}^{1/5}$$

The constants in Equation (10) were evaluated by the method of least squares. The values obtained for C_1 , C_2 and C_3 , listed in Table 3, were all positive thereby validating the surface reaction between an adsorbed carbon dioxide molecule and two dissociated hydrogen molecules as a possible rate-

controlling mechanism. Values of these constants were substituted in Equation (9) and average rates of reaction were calculated and compared with the experimental rates. The rates listed in Column 14 of Table 2 which were computed as described in Calculated Results will be referred to as experimental rates to distinguish them from the rates obtained from the correlations.

Many other surface mechanisms were assumed to be rate-controlling in order to find the one which would best fit the data.

It was found that the rates of methane formation could be correlated satisfactorily for any mechanism which involves the reaction of an adsorbed carbon dioxide molecule and at least two dissociated hydrogen molecules. Equation (9), representing one extreme of the acceptable range of mechanisms, presents the best correlation of the

data. Equation (11), representing the other extreme involving four hydrogen molecules, presents a correlation almost equally good. Use of Equations (9) and (11) to calculate rates of methane formation will be referred to as Methods A and B, respectively.

$$r = \frac{C_1'(p_{CO_2}p_{H_2}^4 - p_{CH_4}p_{H_2O}^2/K_1)}{(p_{H_2}^{1/2} + C_2'p_{CO_2} + C_3')^5} \quad (11)$$

The constants were calculated at each of the other experimental temperatures throughout the range. The logarithms of these constants were plotted versus the reciprocal of the absolute temperature in Figures 27-32. Variation of C_1 with temperature was smoothed by drawing the best straight line through the plotted points. Variations of C_2 , C_1' and C_3' with temperature were smoothed in the same manner. Revised values of C_2 and C_3' were calculated at each experimental temperature by the method of least squares assuming the smoothed values for the other constants were valid. These revised points were plotted in Figures 29 and 32.

In each of these figures, the points lay on a smooth curve which was nearly linear throughout most of the temperature range. Table 3 presents the original and revised values of the constants at each temperature.

The following equations express the temperature variations of the revised constants:

$$\ln C_1 = -26,950/T + 29.8 \quad (500-750^\circ F.) \quad (12)$$

$$\ln C_2 = 1240/T - 2.04 \quad (500-750^\circ F.) \quad (13)$$

$$\ln C_3 = -8670/T + 8.43 \quad (650-750^\circ F.) \quad (14)$$

$$\ln C_1' = -14,700/T + 17.47 \quad (500-750^\circ F.) \quad (15)$$

$$\ln C_2' = 2080/T - 3.63 \quad (500-750^\circ F.) \quad (16)$$

$$\ln C_3' = -6100/T + 3.47 \quad (650-750^\circ F.) \quad (17)$$

where T = absolute temperature, $^\circ R$. Values of C_2 and C_3' should be obtained from Figures 27 and 32 respectively, but Equations (14) and (17), applicable only in the temperature

TABLE 3.—COMPARISON OF CALCULATED AND EXPERIMENTAL RATES

| Space Time (hr. $\times 10^3$) (2) | Average Partial Pressure of Component (Atm.) | | | | | Average Rate of CH ₄ Formation lb. mole / (day) (lb. catalyst) Calculated | | | | | Deviation lb. mole / (day) (lb. catalyst) (Rate. — Temp.) | | | | |
|---|--|------------------|-------------------|-------------------|--|--|--------------|--------------|---------------|-------------------|---|--------------------|--------------------|--------------------|--|
| | | | | | | | | | | | | | | | |
| | (3) p_{CO_2} | (4) p_{H_2} | (5) p_{CO_2} | (6) p_{H_2O} | | (7) r_A | (8) r_B | (9) r_A | (10) r_B | (11) r_{exp} | (12) Δ_1 | (13) Δ_2 | (14) Δ_3 | (15) Δ_4 | |
| 0.000 | | | | | | | | | | | | | | | |
| 0.005 | .1910 | .775 | .0078 | .0210 | | 1.41 | 1.47 | 1.42 | 1.49 | 1.21 | + .20 | + .36 | + .21 | + .28 | |
| 1.460 | .1852 | .739 | .0245 | .0503 | | 1.41 | 1.44 | 1.40 | 1.46 | 1.32 | + .31 | + .18 | + .32 | + .16 | |
| 2.955 | .1739 | .6925 | .0485 | .0860 | | 1.41 | 1.38 | 1.39 | 1.40 | .90 | + .75 | + .72 | + .67 | + .74 | |
| 4.94 | .1589 | .6305 | .0703 | .1406 | | 1.34 | 1.30 | 1.35 | 1.32 | .95 | + .39 | + .35 | + .30 | + .37 | |
| 7.85 | .1340 | .534 | .1110 | .221 | | 1.26 | 1.16 | 1.10 | 1.16 | .71 | + .55 | + .45 | + .39 | + .45 | |
| Temperature—650° F. | | | | | | | | | | | | | | | |
| 0.000 | | | | | | | | | | | | | | | |
| 0.005 | .176 | .751 | .016 | .045 | | 2.26 | 2.04 | 2.24 | 2.02 | 2.10 | + .16 | + .04 | + .14 | + .08 | |
| 1.460 | .166 | .689 | .045 | .096 | | 2.24 | 2.05 | 2.25 | 2.05 | 2.28 | + .08 | + .08 | + .07 | + .02 | |
| 2.955 | .149 | .595 | .087 | .171 | | 2.45 | 2.25 | 2.49 | 2.55 | .97 | + 1.46 | + 1.55 | + 1.51 | + 1.56 | |
| 4.94 | .123 | .469 | .136 | .308 | | 1.71 | 1.96 | 1.61 | 2.02 | 1.19 | + .82 | + .77 | + .69 | + .83 | |
| 7.85 | .094 | .395 | .199 | .599 | | .90 | 1.34 | 1.01 | 1.32 | .79 | + .11 | + .45 | + .33 | + .43 | |
| Temperature—550° F. | | | | | | | | | | | | | | | |
| 0.074 | .1908 | .608 | .1508 | .0415 | | 2.99 | 2.99 | 2.03 | 2.00 | 2.82 | + .88 | + .83 | + .79 | + .82 | |
| 1.424 | .1988 | .498 | .308 | .1366 | | 3.31 | 2.82 | 2.39 | 2.59 | 2.81 | + .50 | + .39 | + .43 | + .32 | |
| 2.878 | .1986 | .367 | .322 | .322 | | 1.55 | 1.95 | 1.67 | 2.04 | 1.59 | + .03 | + .37 | + .09 | + .46 | |

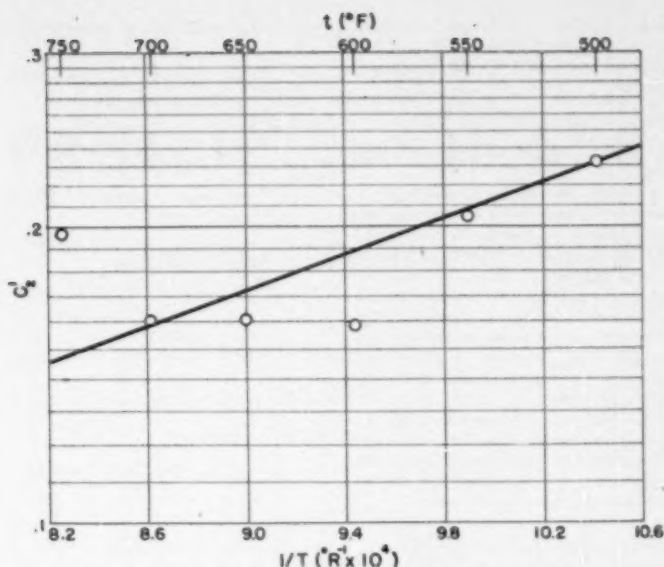


Fig. 31. Variation of rate-equation constant C_e' with temperature.

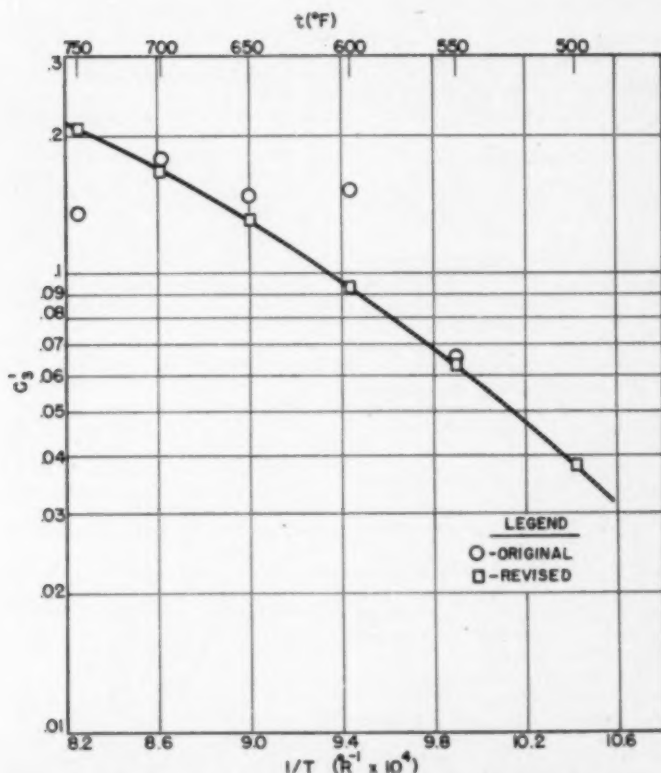


Fig. 32. Variation of rate-equation constant C_e' with temperature.

range of 650-750° F., are presented to make possible an approximate determination of enthalpies and entropies of activation and adsorption. These thermodynamic quantities can be correctly evaluated only if the assumed mechanism is valid and if the catalyst activity is independent of temperature.

Since C_a and $C_a' = 1/K_{H_2}$ for their respective mechanisms by previous definition, the following expressions can be written from Equations (14) and (17), respectively:

$$\ln K_{H_2} = 17,340/T - 16.86 \quad (18)$$

$$\ln K_{H_2} = 12,200/T - 6.94 \quad (19)$$

A comparison of Equations (18) and (19) with the well-known relation between an equilibrium constant and temperature

$$\ln K = \frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (20)$$

permits the evaluation of the standard enthalpy and entropy charges of hydrogen adsorption for methods A and B respectively. A similar procedure with Equations (12) and (15), and (13) and (16) gives the standard enthalpy and entropy charges of carbon dioxide adsorption and the enthalpy of activation. These values are presented in Table 4.

Rates of methane formation were calculated by Methods A and B using the revised values of the constants and these rates, the original calculated rates, and the experimental rates are compared in Table 5. Columns (7) and (8) present the original rates calculated by Methods A and B, respectively, and columns (9) and (10) list the rates calculated using the revised values of the constants. Columns (12-15) present the deviations of these rates from the experimental rates which are listed in Column (11). The average deviations show the revised rates to be equally good as the original calculated rates. The average deviation of the revised rates from the experimental rates varies from ± 0.15 and ± 0.17 for Methods A and B, respectively, at 500° F. to ± 0.40 and ± 0.50 for Methods A and B, respectively, at 750° F. The maximum experimental rate varies from 1.06 at 500° F. to 5.60 at 750° F. Rates and the deviations are expressed in lb.moles/(day) (lb.catalyst).

Rate equations were integrated over the experimental conditions of the various runs in order to reproduce the partial pressure of methane in the gases. Values calculated by Methods A and B are plotted in Figures 2-25. The average durations of the calculated partial pressure of methane vary from ± 0.0037 to ± 0.0107 atmospheres, an excellent

check in view of the cumulative effect of error in the rate equations.

Considerably more data have been obtained for this study than are usually obtained from an investigation of this type, and yet several equations each representing a different mechanism, can be written which will reproduce the data to a degree of accuracy comparable with that of the methods of analysis used. The accuracy of these measurements compares favorably with that obtained in similar investigations reported in the literature.

The theoretical correlation advanced the idea of a reaction between an adsorbed carbon dioxide molecule and at least two dissociated hydrogen molecules adsorbed on four adjacent active centers. From a statistical point of view this is an unlikely event and thus Equations (10) and (11) serve as most unattractive theoretical possibilities in spite of the excellent correlation of the data which they provide. In view of the accuracy of the experimental data, it would appear that the theoretical approach used offers only faint hope of arriving at an analysis of mechanism from kinetic data alone.

The equations obtained by the theoretical treatment are rather cumbersome and for many engineering purposes, empirical relations of simpler form probably can be used to reproduce the data, although extrapolation of such relations is not recommended. For example, one reviewer reported excellent success in correlating the data at 750° F. by means of an equation of the form

$$r = k_1 p_{CO_2} p_{H_2} - k_2 p_{CH_4} p_{H_2}^2 \quad (29)$$

At the present time, data on the $CO_2-CO-H_2-CH_4-H_2O$ system are too meager to justify extensive empirical correlation. The course of future research would appear to be a thorough investigation of the equilibrium adsorption for each component and for various mixtures of components including precise measurements of the enthalpy changes accompanying the adsorption. This adsorption-equilibria study should be supplemented by additional rate data obtained under pressure with feed mixtures already studied at one atmosphere. Rate data should also be obtained at several pressures for feed mixtures of other components such as carbon monoxide and water.

Conclusions

When carbon dioxide-hydrogen mixtures are passed over a reduced nickel catalyst in the temperature range 500-750° F., the following over-all reaction occurs:



Small amounts of carbon monoxide which at no time exceed 3.5% are also formed, apparently by the following reaction:



The carbon formed under these conditions is negligible.

The surface reaction appears to be rate-controlling. Rates of reaction can be correlated for any mechanism which assumes that the major resistance to the over-all process is offered by the reaction of an adsorbed carbon dioxide molecule with at least two adsorbed hydrogen molecules. The assumption that the adsorbed hydrogen is dissociated results in better agreement with the experimental data.

The following two equations for the rate of methane formation represent the extremes of the range of acceptable mechanisms and reproduce the experimental data almost equally well:

$$r = \frac{C_1 \left(p_{CO_2} p_{H_2}^2 - \frac{p_{CH_4} p_{H_2}^2}{K_1} \right)}{(p_{H_2}^{1/2} + C_2 p_{CO_2} + C_3)^8}$$

$$r = \frac{C_1' \left(p_{CO_2} p_{H_2}^4 - \frac{p_{CH_4} p_{H_2}^3}{K_1'} \right)}{(p_{H_2}^{1/2} + C_2' p_{CO_2} + C_3')^9}$$

Acknowledgment

This research was sponsored by the Michigan Gas Association.

Notation

C_1, C_2, C_3 = rate equation constants for correlation by Method A

C_1', C_2', C_3' = rate equation constants for correlation by Method B

c_A = concentration of adsorbed A per unit mass of catalyst

D = deviation of calculated value from experimental value

H = enthalpy

ΔH^\ddagger = standard enthalpy of activation

K_1 = equilibrium constant of reaction 1

K_1' = surface equilibrium constant of reaction 1

K_A = equilibrium adsorption constant for component A

k = forward-reaction velocity constant

k' = reverse-reaction velocity constant

k_A = adsorption velocity constant

k_A' = desorption velocity constant

N = number of catalyst pellets

P = total pressure

p_A = partial pressure of component A at the gas-solid interface

r = rate of methane formation

r_A = rate of adsorption of component A

r_A = rate of methane formation calculated by Method A using smoothed rate-equation constants

r_A' = rate of methane formation calculated by Method A using unsmoothed rate-equation constants

S = entropy

S_r = space velocity

s = number of active sites adjacent to any given site

T = absolute temperature, °R.

t = temperature, °F.

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PRINCIPLES OF SPRAY DRYING

PART II—ELEMENTS OF SPRAY-DRYER DESIGN†

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IN Part I, the basic principles underlying spray drying were considered. Part II will consider the design aspects of spray drying.

1. Establishing Operating Variables

Design data on certain variables of spray-dryer operation must frequently be determined by experimental tests before the final design of a spray dryer can be made. In general, the design of the dryer chamber, the selection and design of the atomizer, and the product recovery system will be governed by the characteristics of the solution or suspension to be dried and by the desired characteristics of the dried particles. These considerations imply that a spray dryer must be carefully designed to fit a pattern of mechanical sequences required in the atomizing and drying of a specific fluid, and the collection of the powder produced therefrom. No single spray dryer is sufficiently flexible or versatile to accommodate the variety of foods, soaps, and chemicals dried by this method. Where a product manufacturer already possesses or inherits an existing spray dryer there are modifications in construction or operation that may, within limits, adapt the equipment to the handling and production of the desired powder. However, whether adapting an existing dryer or designing a new one, the design engineer should follow certain logical paths which will permit intelligent modification of an existing spray dryer or rational selection and design of a new one. In general, the first path to follow is that leading to an accurate development of design

data on the variables of operation involved. Usually, the most important variables for which design data must be established are:

1. Nature of the product desired; i.e., whether a fine or a coarse product is required, and whether a bead-like particle is desired.
2. Method of atomization to be employed
3. Concentration of the feed
4. Drying temperature
5. Need for cooling with secondary air
6. Method of product removal
7. Dust recovery

It is customary to establish each of these variables from experimental tests, and the factors influencing their determination are considered below.

Nature of the Product Desired. A given material can often be spray-dried to particles of different sizes, such as spheres or spheroids that may be hollow or solid, and agglomerates thereof, or the particles may consist of shattered spheres with the appearance of eggshells (Fig. 6, Pt. I). The determination of whether the product is to consist of hollow or solid spheres is fundamental in deciding the type of dryer to be selected and the techniques of its operation. Hollow spheres can be attractively beadlike and usually have large volume for a given weight; i.e., low bulk density, factors that may contribute to sales appeal. Solid spheres, because of the relative absence of trapped gases, give a higher bulk density. Agglomeration is generally fortuitous, although not without merit for certain types of products. Shattering of spheres is usually unintentional when it occurs within the dryer system; however, some manufacturers later resort to milling of the particle as a means of increasing bulk density.

Particle Size and Size Distribution. These are important because they influence bulk density, solubility, color, free-flowing characteristics, segregation and dustiness. Colored particles that are large and dense tend to look darker and more vivid than small particles of identical compositions and they tend also to be more free-flowing. A wide range of particle sizes can result in sifting and segregation that ultimately causes the appearance of a slack-fill. Creation of dust by ultrafine atomization or shattering of particles imparts a sneezing hazard to laundry products and a handling nuisance to other products. It is easier with most spray dryers to produce a product with a wide range of particle sizes than one with the particle-size range within a narrow band. Only in the production of small particles is a narrow size range usually obtainable directly from the atomizer. It is usually easier to produce hollow large particles with most materials than it is to produce solid large particles.

It is important to understand how the selection of a fine or coarse product affects dryer design. If the spray dryer is designed to produce a fine product, in general it is seldom possible to modify the dryer to produce a coarse, nondusty product. On the other hand, a spray dryer which is intended to produce a coarse product will usually be much larger and costlier than the dryer designed for a fine product. The decision as to a coarse or fine product is usually made by the sales department of the manufacturing organization. It is important, therefore, that such a decision is made with the realization of a spray dryer's inflexibility and the difficulties involved if product changes are required later.

† Part I of this paper, *Fundamentals of Spray-Dryer Operation*, appeared in the October issue of C.E.P. page 501.

Bulk Density. This is one of the most important properties of the product to be studied and determined prior to design. Selection of this property usually depends on economic factors such as type and cost of containers, shipping costs, and marketing requirements. The principal factors influencing bulk density and its control are:

a. **Colloidal Characteristics.** Assuming that spray drying is to be carried out with aqueous solutions or suspensions, there are two general classes of solutions or suspensions that differ markedly in the type of powder produced under certain conditions and follow similar behavior under others. Materials having distinct hydrophilic colloidal properties are in one class, and those of the nonhydrophilic type (but not necessarily hydrophobic) are in the other. Gelatin, soap and sodium silicate are examples of the first class and coffee extract, milk, pigments, and inorganic salts are typical of the second. In order to predict the type of particles produced by spray drying, it is useful to realize in which of the two categories the material to be dried belongs. Thus, drying conditions that produce large particles and low bulk densities with hydrophilic materials might produce small particles and high bulk densities with nonhydrophilic materials.

b. **Solids Concentration.** Solutions of hydrophilic materials generally exhibit a large increase in viscosity with increase in solids. Under most conditions of spray drying such materials, powders of low bulk density are produced, and the bulk density decreases as the solids concentration (and, accordingly, viscosity) increases. Solutions made with gelatin having a gel strength of 125 Bloom or higher are difficult to spray dry at concentrations above 12% solids without obtaining an impracticable voluminous product. Irish moss, a seaweed gum used by the food and drug industry, cannot be spray dried to a satisfactory powder from solutions containing more than 3% solids. A. E. Siehrs (27) describes a process wherein 90% of this gum solution is spray-dried to a powder which is mixed with the remaining 10% of the solution to form a dough. The dough is then extruded and dried in a tunnel. The dried extrusions are milled to produce a dense powder that has good wettability and solubility.

Important exceptions to the above tendencies of hydrophilic materials are soaps and detergents which show a marked increase in bulk density with increase in solids concentration. In fact, increasing the solids content is an important method of increasing the bulk density of spray-dried detergents.

In general, for nonhydrophilic materials such as tannin extract, coffee extract, milk, inorganic salts, pigments and dyes, etc., an increase in solids content will increase the bulk density of the product.

c. **Solution Temperature.** Increasing the temperature of most solutions,

both hydrophilic and nonhydrophilic, tends to increase the bulk density of the spray-dried powder. This has been found to hold true especially for coffee extract, and for soap (12).

d. **Occluded Air.** Air or other non-condensables trapped in the fluid being atomized tend to decrease the bulk density of the powder. Aeration may be accomplished intentionally or accidentally during agitation of viscous or concentrated solutions in an open vessel just prior to spray drying. With simple disk-type or spoke-type rotary atomizers such air may remain trapped in the fluid and contribute to the formation of hollow, gas-filled particles. With more complex centrifugal atomizers, occluded air may be removed from the fluid if the action is foam-breaking, or more air may be introduced if the atomizer acts as an air pump especially when running below its fluid pumping capacity.

With pressure nozzles, the compression and expansion during atomization of the fluid undoubtedly releases some of the air, but a counteracting tendency is a lowering of the fluid temperature, with an increase in viscosity under the action of rapid water evaporation.

The net effect of occluded air is to decrease the bulk density. This is due in part to entrapped air forming a particle with a cellular structure, as shown in Figure 6. (See Pt. I.) Occluded air is not the principal reason for the formation of hollow particles. Ladisch (11a) has discussed the effect of occluded air on particle size and shape.

e. **Temperature of Drying Air.** Bulk density is influenced significantly by the temperature of the air that the spray first encounters, high air temperatures tending to produce low bulk densities. This is attributed to the fact that hot air promotes "balloon formation" due to rapid drying and case-hardening of the particle exterior and to subsequent expansion of trapped moisture. If high bulk density (or low specific volume) is required, or if solid particles are required, the system should be designed so that the hottest air does not encounter the freshly formed spray. In certain food dryers where air is indirectly heated by steam, the inlet air temperature does not exceed 350° F. and, accordingly, the hollow spheres formed may not be of as great an initial diameter as those produced by chemical dryers where air is heated to as high as 1000° F. It is not uncommon to find instances where the pressure of trapped vapor has been sufficient to blow holes through the shell of the hollow particle. This may be observed in Figure 6, Pt. I.

f. **Relative Direction of Air Flow.** Without sacrificing thermal efficiency and capacity, it is possible in spray drying some products, such as soap, to introduce hot air at the bottom of the tower and to spray downward from the top or sides of the tower; i.e., countercurrent to the air. Since the spray of wet particles encounters partly cooled, moist air,

the product consists predominantly of solid spheres or granules, and the bulk density is higher than in concurrent drying. Agglomeration is common, in countercurrent drying because as the falling particles dry during their descent, some attain a lower relative density than others with respect to the air and are either carried upward and collide with and adhere to wet particles, or are overtaken and collide with heavier, faster-falling particles. The product from countercurrent dryers has a less uniform appearance and is less headlike than that obtained from a concurrent dryer. Since the driest particles fall through the zone of hottest gases, this type of dryer is not well suited to most heat-sensitive powders, since inlet temperatures would have to be low resulting in large and expensive dryers. A severe disadvantage of countercurrent flow is the explosive hazard created by a concentration build-up of dust at some point in the tower where particles of the proper weight are held in suspension by the countercurrent air flow. Further, considerable wall build-up occurs in countercurrent dryers.

g. **Powder Milling and Size Distribution.** Any means of filling voids or interstices of spray-dried particles with smaller particles or fragments will tend to increase bulk density. Thus, atomization to furnish a wide-size range would produce a denser but dustier product. Milling of part or all of the product as it leaves the tower is sometimes resorted to in order to increase density. Helical or cut-flight conveyors are sometimes used for shattering large hollow spheres.

h. **Additives.** In spray-drying soaps and detergents it has been found that the addition to the feed of inorganic salts, such as sodium chloride, will increase the bulk density of the product.

Selection of the Method of Atomization. Selection of the atomizer best suited to the problem may automatically determine the type of spray dryer to be used, as well as the manufacturer of the dryer. Determination of the best method of atomization requires experimental tests which in turn require suitable test equipment. Unfortunately, suitable experimental equipment for comparative spray-drying tests by the usual three methods of atomization is seldom available in one place, if available at all. As a result, tests are generally made on whatever equipment may be available with no opportunity for comparative tests.

It is difficult to state reasons unequivocally for choosing one atomizer in preference to another prior to actual atomization and drying tests. It is believed that a majority of spray-drying problems can be handled equally satisfactorily by a pressure nozzle, a two-fluid nozzle, or a spinning-disk atomizer.

Too much mystery and unfounded claims, and not enough basic facts have been associated with atomization in spray drying. Not until comparative performance tests have been made on all three types of atomizers can there be established any rational basis for the selection of one atomizer for spray drying in preference to another. Such comparative performance must eventually consider capacity per horsepower input related to the degree and uniformity of atomization accomplished. Fundamental properties for comparison were proposed in Part I of this paper.

At the present time, certain rules of thumb are followed in selecting, evaluating, and comparing the three types of atomizers. Some of the more salient characteristics of atomizers, which are pertinent to their selection and operation are indicated below:

a. *Pressure Nozzles.* Some of the more significant properties of pressure nozzles have been itemized.

1. These atomizers have been divided into the two general classifications of hollow-cone and solid-cone nozzles. In the former, the fluid emerges from the nozzle as a film moving centrifugally around the periphery of the orifice, from which it breaks up into a hollow-cone spray. Rotary liquid motion prior to emergence from the nozzle is claimed to be necessary for good atomization with most pressure nozzles. This motion is obtained by means of tangential inlets, specially grooved cores, or spinners, and other devices.

2. Nozzle capacity is usually expressed as proportional to an empirical constant times the square root of the pressure, and with water as a reference substance various factors, usually less than one, are applied for solutions and slurries. A single pressure nozzle may handle as much as 10,000 lb. of feed/hr. for spray drying.

3. Any nonuniformities or roughness of the orifice destroys the uniformity of atomization. The fact that nonuniformity and roughness can occur by uneven wear due to erosion is a principal disadvantage of these nozzles. Small orifices on the order of 0.025 in. in diameter may become oval and increase a full

drill size in a single day's operation. Larger orifices show far less erosion after a similar period of time.

4. It is generally preferable to spray-dry with a single nozzle, primarily for convenience and labor-saving, and only secondarily because of nozzle parts replacement, which are a small factor in the over-all economics. However, some established spray dryers make use of multiple nozzle arrangements to good advantage. In general, multiple nozzles are used when straight-line, parallel air flow is employed, and a single nozzle is generally used with rotary and countercurrent flow, although soap dryers generally employ multiple nozzles with countercurrent flow. The case for single versus multiple nozzles, however, is far from a settled issue.

5. Pressure nozzles are somewhat inflexible from the standpoint of their inability to be easily adapted to meet changes in operating conditions.

6. Spray towers designed for pressure nozzles rarely can be converted for use with disk atomizers.

Larcombe (15) and Doble (4a) reported performance characteristics and design methods for pressure nozzles.

b. *Spinning Disk Atomizers.* These atomizers have found widespread

application in the chemical industries. Some of their more important operating characteristics are given here.

1. Disk atomizers can handle practically all fluids that can be sprayed with nozzles, and in addition they have been used successfully to atomize thick slurries and crystal suspensions. Capacities of 30 gal. of feed/min. can be atomized by a single disk.

2. In addition to the flexibility of accommodating capacity changes and variations in fluid characteristics, such as viscosity and concentration, the disk atomizer in many cases makes possible moderate adjustment in bulk density by a variation in the speed. By varying the disk diameter and/or the type of disk, particle size changes can be effected and auxiliary operations performed such as foam-breaking, homogenizing, and mixing of ingredients. For example, in a certain manufacturing operation, the two principal ingredients cannot be blended until the instant before spray drying, otherwise they will react. Therefore, solutions or suspensions of each ingredient are fed simultaneously from separate lines in the correct proportions to the disk and are blended and dried. The temperature is maintained low by evaporative cooling, and interaction does not occur.

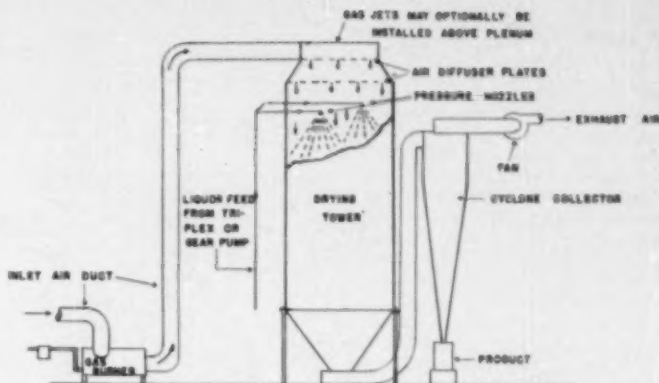


Fig. 7. Vertical Downward Cocurrent Spray Dryer (Zirnia type) with Straightline Air Flow.

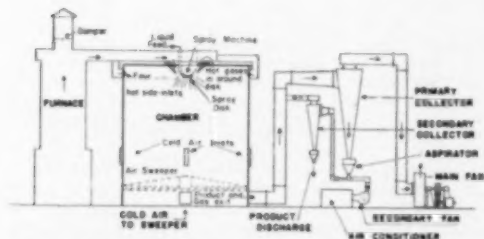


Fig. 8a. Bowen Spray Dryer with Rotary Air Sweeper to Cool and Remove Product. All product leaves with the exit gases.

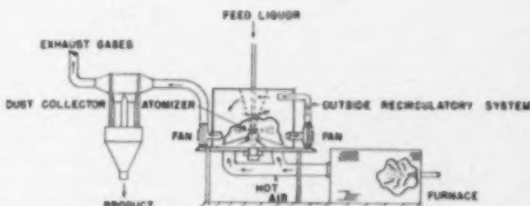


Fig. 8b. Vertical Upward Cocurrent Spray Dryer. (Western Precipitation Corp.)

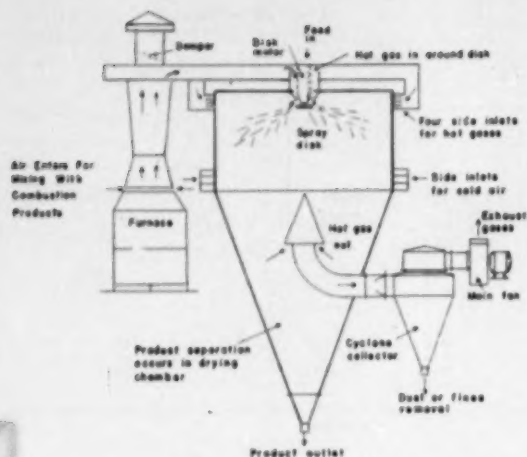


Fig. 9a. Bowen Spray Dryer for Soaps, Detergents and Other Coarse-Particle Products.

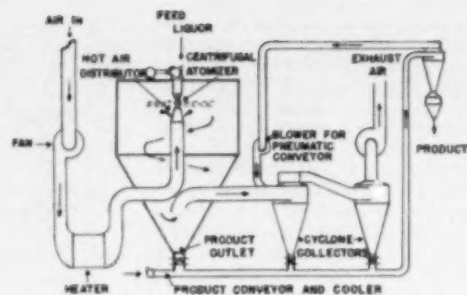


Fig. 9b. Niro Spray Dryer.

3. Disk atomizers may be suspended from the top of a spray tower or may be supported just above the floor of the tower, in which case they are underdriven (See Fig. 8a & 8b). In both cases the flow of hot air must be concurrent, which means downward flow for the former and upward for the latter. Disks are not used, however, in concurrent dryers where the air and spray flow horizontally from inlets through a vertical wall, as in the Merrell-Soule and Rogers-type spray dryers, or as in the Buřlovsk spray dryer.

4. Incrustation on the atomizer disk is more serious than in the case of nozzles, since imbalance of a high-speed disk will not only cause imperfect atomization but also may cause damage to the mechanism and the tower. In general, drying of material on the spray disk usually can be avoided by proper design of the disk and the hot-air inlet.

5. An extensive study of the operation of various types of disk atomizers has been reported in an investigation of drying clay slips by the Bureau of Mines (29).

c. **Two-Fluid Nozzles.** Two-fluid nozzles have probably found least widespread application in spray drying. They have been used primarily for spray-drying soap, and in experimental spray dryers. Some of their characteristics are cited below.

1. A two-fluid nozzle employs a compressible fluid, such as air or steam, to disrupt a liquid into a spray.

2. These nozzles may be classified as either internal or external mixing. The former operates on the ejector principle and the latter uses a gas jet directed at the liquid issuing from an orifice.

3. The two-fluid nozzle appears to be well-suited to the atomization of viscous liquids.

4. Some controversy exists regarding the efficiency of two-fluid

nozzles at high capacities. Unfortunately, no published data are available on the performance of these nozzles at high capacities. Certainly it is true that considerably more energy may be obtained from this nozzle than from a pressure nozzle, but no evidence seems to be available concerning the efficiency with which this energy can be utilized for atomization.

5. Atomization does not appear to be as uniform with these nozzles. This may be due to uneven distribution of the energy of the atomizing fluid.

6. Steam is commonly employed as the atomizing means in large installations. A typical large-scale installation might utilize 100 lb./sq.in. steam to atomize soap with 63% solids and under 40 lb./sq.in. pressure.

7. The relationship between drop-size and gas-liquid ratio for this type of atomizer was presented in Part I of this paper.

Selection of Proper Feed Concentration. Determination of the feed concentration for design purposes can be done only by experimental tests to furnish product for evaluation. The concentration will usually be determined by the following considerations:

1. Effect on quality and dryer operation
2. Effect on bulk density
3. Maximum concentration which can be pumped and atomized, i.e., solution or slurry characteristics
4. Heat load or fuel costs

In general, the tendency should be to select the maximum concentration compatible with these four considerations. The highest possible concentration will usually be determined by the pumping and atomizing requirements, if the bulk density is desired high, and if product

quality is unaffected by concentration. However, there are often quality or operational limitations on the degree of concentration possible with certain materials. Some examples are cited as follows:

Milk: Preconcentrating of milk affects the solubility of the powder made therefrom to only a negligible extent provided that the forewarming and concentrating temperatures do not exceed 160° F. Primarily for viscosity reasons it has not been feasible to concentrate above 40% solids, and most spray drying operations are carried out with milk having under 35% solids.

Irish Moss: Solutions of this seaweed gum are exceedingly viscous and foam badly so that they are difficult to preconcentrate above about 5% solids. They cannot be spray dried above 3½% solids without getting strings and ribbons instead of discrete particles.

Gelatin: Gelatin at gel strength suitable for desserts cannot be spray dried above 15% solids without getting the same impractical result even though it is possible to preconcentrate to 30% solids. Gelatin of glue grade is far less viscous and can be spray dried at the latter concentration.

Soap and Detergents: Soap can be spray dried at practically any concentration sufficiently fluid to deliver to the atomizer, and may contain up to 70% solids (62-66% is average).

Corn Syrup: Corn syrup having 65% solids can be spray dried successfully.

Selection of the Drying Temperature. The drying temperature is usually determined solely by the heat-sensitivity of the product. If the product is completely temperature insensitive, then the limitation on temperature will usually be materials of construction.

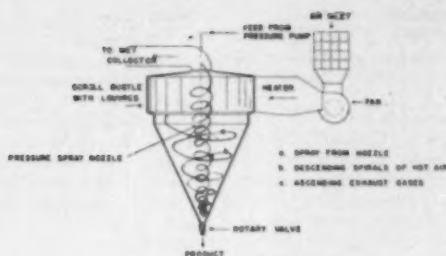


Fig. 10. Combined Cocurrent Countercurrent Spray Dryer with Cyclonic Air Flow Causing Product Separation in Dryer. (Swenson Evaporator Co.)

Need for Cooling Product. Design data on this item can be established only by tests. Those products which require secondary air for cooling after drying are generally thermoplastics, and certain food products containing sugar. Frequently, the need is to maintain the interior surfaces cool to prevent sticking. Careful design is necessary to meet this requirement.

Method of Product Removal. Two principal methods are used for product removal.

1. Product is conveyed from the dryer by the exhaust gases. This is generally the easiest method to design for, and is used chiefly when a fine product is obtained and particle breakage is no objection. Many schemes have been designed for product removal by this method, the usual objectives being, in all cases, to remove all the product so that no dry material remains inside the dryer in contact with hot surfaces.

2. The second is based on the principle of separating most of the product from the gas in the drying chamber, removing the product through appropriate discharge ports, while the dust-laden exhaust gases leave from a second outlet to dust collectors. This procedure is generally followed when a coarse product is desired with a minimum of bead breakage. This is a more difficult design, due to the problems involved in removing the gases without entraining too great a percentage of the product. On the other hand, the classification of fines in the exhaust gas that occurs with this method frequently gives a more dust-free and hence more desirable product. Diagrams of dryers utilizing both types of product removal are shown in Figures 7-10.

Recovery of Dust. Inasmuch as a spray-drying process inherently creates a subdivided material it is evident that the collection of fines and dust is an

important auxiliary operation. The efficiency with which both product and dust can be recovered may make or break the spray-drying operation. Collection systems which may be involved in spray drying are: cyclone collectors, bag filters, and wet collectors or scrubbers. These systems are necessary for the following reasons, and one or all may be found on a spray-drying system:

1. Primary collection of product when the product is conveyed from the dryer by the exhaust gases
2. Recovery of dust from exhaust gases after primary separation in the dryer chamber
3. Recovery of the dust, escaping from cyclone collectors because of the value of the product. Bag filters or wet collectors may be used
4. Scrubbing of exhaust gases from cyclone collectors because the dust represents a nuisance, but collection is not justified on an economic basis

Selection and design of the various collectors is dependent on the nature of the dust involved and its particle size. Procedures for selecting proper equipment have been reported in the literature, and a comprehensive survey of aspects of dust sampling and collection has been presented by Lapple (14).

2. Design and Operating Faults to Avoid

Poor Spray-Gas Mixing. The efficiency of a spray dryer is severely impaired if the spray and hot gases are not efficiently mixed. Efficient mixing implies uniformity as well as speed. It was shown in Part I that the evaporation times in spray drying may be considerably less than a second. Consequently the time required to mix the gas and spray efficiently should be of the same order of magnitude as the drying time, otherwise the benefits from atomization are lost.

In general, the great multitude of dryer designs reflect various attempts to improve the spray-gas mixing. However, many such attempts introduce more serious complications, such as violent eddying and turbulence which tend to promote wall build-up or particle degradation.

Little is known about the complicated air currents in a spray dryer resulting from momentum transfer from spray to gas, sudden cooling of the gas with reduction in volume, and the large temperature gradients between the hot gas and the evaporating drops. If there is imposed on these phenomena, violent air motion to promote mixing, such as high rotational velocities, common in many designs, the resulting flow pattern is exceedingly difficult if not impossible to predict.

At the present time, many types of spray dryers include design features for which claims are made without fundamental data to justify them. There is no conclusive evidence, for example, that a rapidly rotating stream of air entering centrally in a cocurrent dryer should rotate in one direction in preference to another, or in fact that it should rotate at all in some applications. There is no conclusive evidence that air entering the top of a cocurrent dryer should not be distributed uniformly over the top rather than entering at a high ve-

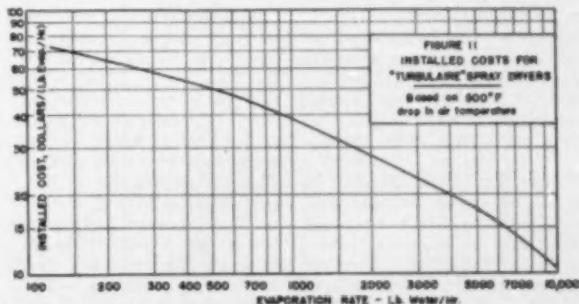


Fig. 11.

locity through a small central zone. There is no conclusive evidence on when hot-air side inlets have real value and when they do not. Some dryers have been designed and installed with such inlets, but have been found to operate better with the inlets closed off. In other words, the complicated flow patterns in a spray dryer are so little understood and actually so exceedingly complex, that a great deal of study must be consumed on the problems of spray-gas mixing, the entrainment of air in sprays, and convection during drying before spray-dryer design can be lifted from its present realm of art and empiricism.

Product Degradation. The designer of spray dryers should avoid passing product through fans or other equipment causing particle degradation. This increases the problem of dust recovery as well as degrading the properties of the product.

Non-Uniform Atomizer Performance. A serious operating fault may be encountered in connection with nonuniform atomizer performance. Nonuniformity of atomization may be of two types: (1) nonuniform particle-size distribution; i.e., the creation of a higher percentage of fine or coarse particles than the usual size distribution function for the spray would predict, and (2) nonuniform weight distribution in the spray pattern.

The first type of nonuniformity will lead either to overheating and degrading of fine particles, or nondrying of coarse particles and sticking of wet material on the chamber. This fault can be avoided by selection of the proper atomizer and by operating the atomizer within the range of design capacity. It is not impossible to have a nonuniform spray develop during operation as a result of erosion, plugging, and incrustation of the atomizer.

The second type of nonuniformity, nonuniform spray distribution, is generally due to faulty atomizer design. With disk atomizers, the feed must be uniformly distributed onto the disk, for no amount of disk rotation will correct nonuniform feed distribution. The effect of such maldistribution is to unbalance the evaporation process, resulting in further complications in air flow and product movement in the dryer. Of course, operational faults, such as plugging, may cause nonuniform spray distribution.

Two other nonuniformities may be encountered to impair atomizer and dryer performance. These are: (1) nonuniform flow of feed, and (2) nonuniform feed concentration. These are obviously operational problems and responsibilities of the product manufacturer. Such nonuniformities are re-

flected in fluctuations in outlet temperature, nonuniformity in final moisture content, overheating or underdrying of the product, and possibly variations in bulk density.

It cannot be overstressed that satisfactory spray-dryer performance depends on steady, balanced or symmetrical air flow into the dryer, uniform or balanced atomizer performance, and a steady uniform feed. Fluctuations in any of these conditions lead to poor dryer performance, wall build-up, and nonuniform product.

3. Spray Dryer Performance

In general, spray dryers operate over a wide range of conditions. They handle dilute solutions of 1% concentration up to thick pastes of 70% solids. They may operate with inlet temperatures ranging from 175° F. inlet to 1400° F. Heat for drying may be obtained from steam, oil, gas, coal, or electricity.

In the food industry, a large percentage of spray dryers employ indirect steam heaters for air heating, and accordingly, the inlet temperature, being limited by the available steam pressure, is below 330° F. This imposes a ceiling on evaporative capacity for a fixed chamber size and air capacity. Steam heating has several advantages, however:

1. It is convenient. When there is sufficient boiler capacity, the use of steam reduces the amount of attention and labor normally needed for a direct-heating system.
2. Indirect heating is cleaner. An auxiliary stack is not required for start-up, and there is no danger of soot getting into the product, such as may occur with incorrect combustion in direct heat systems.
3. It is relatively nonhazardous inasmuch as the temperatures are rarely high enough to cause smoldering of wall products.
4. It is not unduly expensive if low-pressure steam is used for preheating or concentrating the feed liquor, in which cases the consumption may be as low as 1.4 to 2 lb. steam/lb. water evaporated.
5. Use of relatively low temperature inlet air is conducive to the attainment of high density powders.

Another method of indirectly heating air is by passing it through a heat exchanger heated by the flue gases from the boiler house before they are discharged to the atmosphere. A steam heater is installed in the system mainly as a means of regulating the temperature of the hot air, but also can be used when the plant is starting up. Another type of double heating system is one in which steam-heated air passes through a booster heater indirectly heated by fuel oil or gas, or it may mix directly with products of combustion.

Temperatures of 600° F. and higher have been used for drying certain foods

without impairment of quality when care has been taken to avoid by-passing of some of the entering hot gases around the initial drying zone. Such high temperatures are best obtained by mixing intake air with the products of combustion of fuel oil or gas. Evaporative cooling prevents overheating of the particles in the primary drying zone, and the temperature of the particles in this zone is believed not to exceed the wet-bulb temperature of the air. Beyond this zone, when unsaturated surface drying begins (corresponding to the falling-rate period), drying must be continued in a relatively cool atmosphere. Several dryers show the deficiency of having some hot air skirt the initial drying zone to areas where the partly dried particles can be overheated. Where such by-passed air rapidly becomes mixed with cooler air coming from the primary drying zone such danger may not occur, but considerable drying potential may nevertheless be wasted. The time of drying in the secondary zone, which usually comprises the entire chamber volume with the exception of that within about a 3-ft. radius of the atomizer, determines the final moisture content of the powder. When the particle path through the dryer is short, a high outlet gas temperature appears necessary to ensure complete drying. For example, when drying soluble coffee powder in a tower having straight-line air flow, the outlet air temperature had to be in the neighborhood of 245° F., whereas when circulatory air flow was employed the travel path was so extended that the outlet temperature could be reduced to 200° F.

The drying rate in this second period is influenced also by the air humidity. Peebles and Manning (20a) have developed a spray dryer for drying hygroscopic fluids such as corn syrup and molasses and wherein the partly dried particles are suspended in a secondary zone of fresh hot air of lower humidity. The tower is cylindrical with a cone bottom, and the fluid is sprayed from the top by a centrifugal atomizer. Hot air at about 320° F. engages the spray in the primary drying zone and is thereby cooled to about 235° F. Most of the air, carrying about one-third of the powder, doubles back up to ceiling outlets and is withdrawn through air separators. Fresh hot air at 200° F., equal to about 40% of the volume discharged through the ceiling, is introduced at lower levels into the tower. The air discharged from the bottom of the cone at about 190° F. carries the remaining two thirds of the powder to the same main separator. For air entering with a humidity of 1.5 lb. water/100 lb. dry air, the air leaving at both the top and bottom outlets is said to contain approxi-

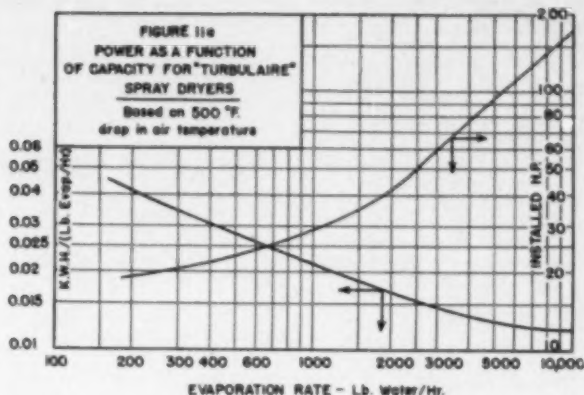


Fig. 11a.

mately 3.5 lb. water/100 lb. dry air.

In most spray dryers having rotary air motion, particularly those of cyclone collector shape, a high percentage of the powder circulates around the walls in descending spirals. Unless the walls are cooled, the product will thereby become heated much more than is necessary for good drying. In one commonly used dryer, milk powder which can be regarded as being at a temperature of 110° F. while suspended in the air is raised to 160° F. while circulating on the wall. With materials such as thermoplastics this is prohibitive, and in the Bowen type of spray dryer (Fig. 8a), for example, a mass of rotating cool air is introduced adjacent to the wall. The cool air may be drawn in through slanted vertical inlets located at several levels along the vertical walls.

Spray dryers have been built in all sizes, from 30 in. in diameter to 40 ft., and 100 ft. high. Materials of construction range from tile and concrete to special stainless steels. Wooden spray dryers lined with coated galvanized sheeting were built for egg drying during World War II.

The heat efficiency of a spray dryer, in general, depends on its temperatures of operation. Approximate expressions for two efficiencies may be written as follows:

1. Overall efficiency, η_o , expressed by

$$\eta_o = \left(\frac{t_1 - t_2}{t_1 - t_a} \right) 100 \quad (23)$$

and

2. Evaporative efficiency, η_e ,

$$\eta_e = \left(\frac{t_1 - t_2}{t_1 - t_a} \right) 100 \quad (24)$$

where $(t_1 - t_2)$ represents the drop in air temperature due to drying only; i.e.,

no heat losses, and t_a and t_s are the atmospheric temperature, and the adiabatic saturation temperature corresponding to t_s , respectively. Evaporative efficiency indicates the degree of approach to saturation reached by the drying air. Overall efficiency indicates the fraction of the total heat input used for drying. The above expressions are convenient approximations in that they do not take account of the variation in humid heat capacity of the air.

It is evident from Equations (23) and (24) that if the outlet temperature is maintained constant, the efficiency of a spray dryer increases with increase in inlet temperature. This says nothing about the possible reduction in dryer size and investment with increased temperature.

The recovery of heat from the exhaust gases of spray dryers has always been a prime concern of the engineer.

With dryers having an evaporative capacity of 1000 lb. water/hr. it is not unusual to discharge 1,000,000 B.t.u./hr. to the atmosphere. The utilization of exhaust hot air for preconcentrating is an established practice for improving heat economy, and serves the additional purpose of scrubbing out and recovering suspended solids. There are several European spray dryers that utilize preconcentrators, and in this country the Swenson spray dryer (Fig. 10) utilizes the recovery of heat by preconcentration. The over-all steam consumption of the Swenson spray dryer for drying milk is claimed to be 1.5 lb. steam/lb. water evaporated. This includes the steam used for preheating the milk in the preconcentrating stage.

Spray dryers are generally controlled by means of the outlet gas temperature. This temperature is extremely sensitive to changes in feed rate, air rate, and inlet air temperature. Further, any unbalance in these variables is noted quickly at the outlet temperature, since the average throughput time in a spray dryer is a matter of seconds. Generally, the feed rate is controlled by the outlet temperature, since it is unusual to encounter variations in air flow and inlet temperatures, and more difficult to control these variables.

4. Spray Drying Economics

Two principal items of cost are associated with spray drying, as with all drying operations, namely, (1) investment cost, and (2) operating costs. In Figures 11 and 11a are shown typical investment and power costs for Western Precipitation spray dryers. These dryers are generally designed for high speed disk atomization to produce a product of particle size less than 325

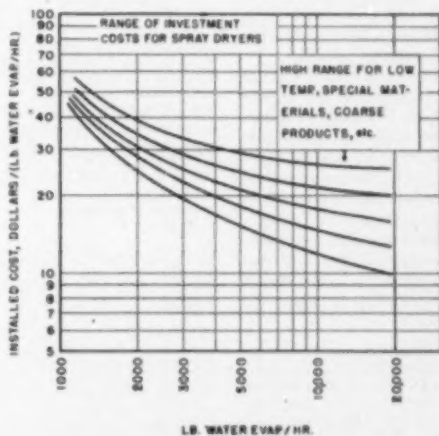


Fig. 12.

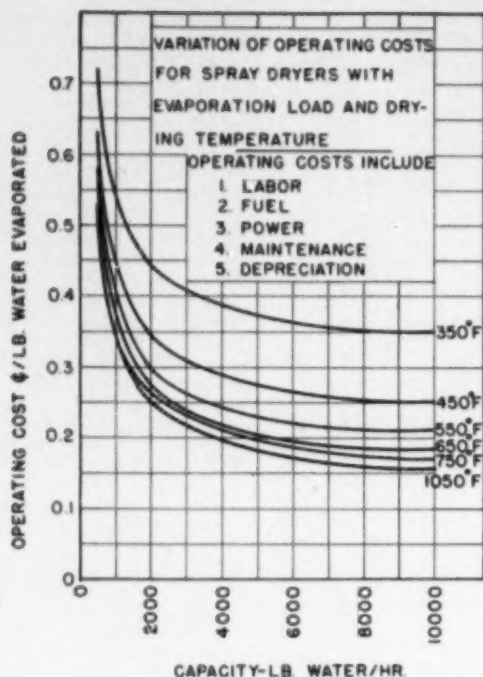


Fig. 13.

mesh. Further, the investment costs are based on dryers operating at an inlet temperature of 750° F. and an outlet temperature of 250° F.

Figure 12 shows a range of investment costs typical of prices encountered on the market today. The upper limit of the range is encountered in low temperature operation, coarse product requirements, and special materials of construction.

The second important cost consideration is operating costs. Generally, operating costs include (1) labor, (2) fuel, (3) power, (4) maintenance, (5) depreciation. Naturally, other cost items such as overhead, royalties, etc., may enter, depending on the plant involved, type of operation, and cost accounting procedures. However, the above items, when evaluated, give a reasonably good index of the cost to evaporate water in a spray dryer.

Figure 13 shows how the operating cost of a spray dryer varies with capacity, and temperature. These curves are based on the following data and assumptions:

- Labor:** It was assumed that one man can operate a spray dryer at an hourly rate of \$1.50. Obviously, labor charges will vary according to the plant, the operations involved, and the methods of allocating labor charges. Since labor was assumed constant regardless

of dryer size, the curves may be readily changed by a constant factor for other labor charges.

- Fuel:** This was estimated from the weight of air to be heated to the inlet temperature from an initial temperature of 50° F. The cost of fuel was taken as \$0.12/gal. for No. 2 fuel oil with a heating value of 136,000 B.t.u./gal.
- Power:** Power was considered to be composed of two items, power for air handling, and power for atomization. Power for air flow was estimated on the assumption of an over-all pressure drop of 8 in. H₂O, and a 60% fan efficiency. Atomizer power was estimated from Figure 4, for disk atomization, assuming a constant value of $N_r = 2500$. A power cost of \$0.01/kw-hr. was used.
- Maintenance and Depreciation:** Maintenance was assumed to be 3% and depreciation 10% of the total installed cost. A 7000-hr. year was assumed, and installed costs were obtained from Figures 11 and 12, taking account of the different temperatures of operation.

In addition to the above cost, the product manufacturer must include the following cost items:

- A minimum of 2% product losses as dust, and wall deposit
- Overhead and overtime labor costs
- Insurance, taxes, and royalties, if any

In the food industry, spray-drying costs are generally higher than the costs

given in Figure 13. This is due to the following reasons:

- Fuel costs are generally higher because indirect heat is usually required.
- More labor is generally required because clean-up problems are more severe and cleanup must be done frequently.
- Plant investment is generally high for the low production rates usually involved.
- Feed concentration is frequently low resulting in greater evaporation loads.
- Food spray dryers are generally used a smaller number of annual hours than chemical spray dryers.

All costs have been based on the pounds of water evaporated per hour. This is because the range of feed concentrations can be so great that many additional curves would be required to cover a concentration range as well. The cost figures given can be converted to dollars per pound of product by multiplying the values on the curve by the value of pounds of liquid per pound of dry solid in the feed. This assumes that feed concentration has no effect either on the cost figures or on the assumptions used in preparing the curves.

It is apparent from the trends in Figure 13 that the higher the capacity the lower the cost per pound of water evaporated or pound of product. At low capacities, the major cost items are labor and fuel. At high capacities, the major cost item is fuel, with investment and maintenance combined being somewhat less. These curves can be used to show the economy to be expected from large-scale spray dryers compared with other dryers which require multiple units to achieve the desired production rate.

Notation

For Notation see Pt. I, page 508.

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Discussion

L. B. Kuhn (Firestone Plastics Co., Pottstown, Pa.): Based on your previous

experience in the handling of latices or emulsified solids, which is somewhat shock-sensitive, is there any data to support the use of a nozzle instead of a wheel or vice-versa?

W. R. Marshall: There is a good deal of patent literature on that matter. As far as I know there is no particular reason for choosing one over the other.

I believe there have been many attempts made to use a two-fluid atomizer. The problem, as I understand it, is to prevent stressing the material so that it does not coagulate prior to atomizing.

I should like to throw that question open, because I see there are a number of men here who may have had experience with this problem.

W. S. Bowen (Bowen Engineering Corp., North Branch, N. J.): The difference between the two latices would come into this classification. In one case it was dried satisfactorily on a pressure nozzle; in the other case it could not be. The reason was not in the nozzle, however; the reason was in the thermoplasticity of that particular latex after leaving the nozzle.

W. R. Marshall: Regarding the matter of selection of atomizers, it is something that appears to be controversial. We find that it cannot always be stated unequivocally that a nozzle should be used in preference to a disk or vice versa. I do not think the question can be answered that simply. Possibly Mr. Seltzer can contribute something on this.

E. Seltzer: I am not acquainted with a spray drying of latices, but I know a comparable case. In the spray drying of albumen the denaturing occurs within the atomizer, and the denaturing occurs within any atomizer that I know of.

Information has just been released on a new atomizer which was developed in Germany during the war. It is available through the P.B. Reports. The author is Kurt Edeler. The device he uses is comparable to a two-fluid nozzle. But the material is fed to the nozzle, adjusts its own hydrostatic pressure, and is atomized under conditions that produce the least disruptive effect upon the fluid.

Anonymous: Would you care to comment on the mechanism of the formation of hollow particles?

W. R. Marshall: One generally accepted explanation is that materials such as soap or gelatin, that is, those which tend to form a dry surface or case-harden rapidly, form hollow spheres. This is due to the fact that the moisture which is entrapped when this case-hardening effect occurs, vaporizes and

expands the particle in such a manner that a hollow space remains once the vapors have removed themselves.

Now, the manner in which the vapors leave the particle can be violent or gentle. You saw some pictures of particles in which violent vapor removal occurred. The resulting blow holes through the particles were evident.

There have been other explanations for hollow particles. I believe one explanation has been called the "waving flag" theory. I do not wish to dispute or endorse it. I believe that there is some evidence for the first explanation. The second one may occur, but I am sure it may have to be under special conditions.

Anonymous: About those black spots you referred to: We noticed them on some products and we have no explanation for them. Has Mr. Seltzer an explanation?

E. Seltzer: I wondered, when first seeing the black spots, whether that was due to the cover spots upon the particles. However, Dr. Marshall pointed out to me that some of the black spots occurred on the sides.

I have no explanation, other than that some heavier ingredients may migrate to the outside and the particle, being spun as it first leaves the atomizer, has not yet set.

R. H. Hemmerich (Shell Chemical Corp., Martinez, Calif.): In the practical application of spray drying, the final product is an important consideration. If you end up with a product, say, that has too low a bulk density, is the basic consideration a change of the atomizer, or what would it be?

W. R. Marshall: You can change several operating variables to change bulk density.

R. H. Hemmerich: What is the theory back of it?

W. R. Marshall: That is a moot point. I do not know whether I can cite theory, but I think it is possible to cite a number of operating conditions which can be considered.

Bulk density is affected by a number of things. I think possibly one of the most significant, as you no doubt have found out, is feed concentration. As you increase the feed concentration, the bulk density tends to decrease. As the temperature of the drying air increases, the bulk density tends to decrease.

An important factor affecting bulk density is the air flow. Once a spray dryer is installed, of course, this is not too easy to change. In countercurrent dryers in which the hot air contacts dry product first, you do not get the sudden

puffing and consequent lowering of the bulk density that occurs when the wet pack suddenly contacts the hot gas.

Solution temperature seems to have an effect, also. I believe if you increase the solution temperature the density can be increased. Is that right, Mr. Seltzer?

E. Seltzer: Yes.

W. R. Marshall: I want to add one more thing, one precaution. In preparing a feed slurry for spray drying make sure that the feed is completely deaerated. If you have any air trapped in the feed mixture, the effect of all these other factors to increase density may be nullified. In other words, if you increase solids content, but in so doing air is entrapped while mixing the feed, all the benefits of increasing solids may be lost. Therefore, you want to be quite sure the feed mixture is deaerated.

The other ways of increasing density are mechanical. You can grind your product, for example.

Anonymous: I'd like to ask Mr. Friedman if, in the drying of porous material, you get a sudden sharp break or a sudden rise in the dryer instead of a gradual rise throughout?

S. J. Friedman (Du Pont Co., Wilmington, Del.): As near as we can tell from all the materials that we have studied, and we have studied porous materials as well as granular materials, we get this flat, level portion of the curve where you apparently do the major portion of your evaporation. Although it is never a sharp rise, it is a sharp inflection point towards the end where your moisture content is reduced to a low value.

This temperature, incidentally, is not always the wet bulk temperature. For some reason, which we do not as yet understand, particularly with cold current drying it may be from five to ten degrees above the wet bulk temperature. And it will stay at that temperature for a substantial period of the drying period.

Anonymous: Do you think it is because the surface temperature is moistened off?

S. J. Friedman: That is what we believe.

Anonymous: In the study on rotary drying, as I recall it, you made the assumption that the hold-up time is sufficient, and that moisture is carried by capillary action and diffusion to the surface. Therefore, the surface-drying phenomena where the length of time was always adequate, developed.

Now, in the spray-drying processes that were discussed, most of those represented might be termed "instant" or "flash" drying processes, where there is

an extension of plant in spray drying, and larger particle sizes, where the drying time becomes important. That would be a function of the particle size.

Did you give any attention to particle size in the rotary drive, since it would affect the hold-up time if the particles become larger?

S. J. Friedman: Well, that is right. The larger the particle the longer it is going to take to remove the diffusional moisture from within. In most cases the particle sizes that we studied were four-mesh or less, in size, and for particles with sizes up to four-mesh we noted that apparently the length of time that the material was kept in the dryer with the hold-up of 2%, was adequate to allow for diffusion of the material, outside of the particles.

Now, in spray drying and in flash drying that is a highly important field. The materials that you have, with a large quantity of diffusional moisture within, generally cannot successfully be flash-dried to a low moisture content. Likewise, you run into the same trouble in spray drying.

W. R. Marshall: A rotary dryer, it might be well to point out, is a rather flexible unit and usually operates within a hold-up range. Thus, the matter of drying time is something you have some control over.

It is not like a spray dryer where the material passes through uniformly in a matter of seconds. In a rotary dryer the drying time is measured in hours and is a function of rate rotation, rate of feeding, slope, and other factors that can be varied. If a rotary dryer is designed for certain operating conditions, it is possible to change those conditions to increase or decrease the hold-up time, and thereby handle any variations in feed rate or feed properties, e.g., variations in particle size. I believe that in the design of rotary dryers one does not design for a specific drying time. Rather the dryer size is estimated on the basis of heat-transfer rates and air velocities. Then the rate of rotation and dryer slope are specified to provide a given holdup. The holdup might go from 3% to 10%. That is a pretty good range to work in, since it permits a variation in contact times or hold-up times by a two- to three-fold range.

S. J. Friedman: That is an interesting point. It happens that the studies on rotary dryers have been limited to a special case in which, by the nature of the equipment, the hold-up time is always adequate. However, when you go to the other extreme, your spray-drying equipment, your hold-up time is usually negligible—unless you keep the particle size down so that you have the equip-

ment for flash or in-tant drying—so you immediately have a problem.

When you get into particle sizes of more than 100 μ , or a thousand, practically all of these spray-drying units that were represented would run into difficulty in giving the desired dryness. Perhaps some of the spray-drying experts will agree, that when you get into the larger particles sizes you have to begin to design your spray dryer with a holdup or height much greater than anything represented—beginning up around 75 or 100 ft. high.

W. R. Marshall: You are making a significant point. In the selection of a spray dryer, a primary decision must be made between a fine product and a coarse. My contention is that on the basis of this selection you determine pretty much the size of your dryer and many other factors.

That brings up the relative inflexibility of the spray dryer. I did not mention it, but the thing we have concluded is that in case a spray dryer is built for one set of conditions, we do not believe it is too easy to change it for radically different conditions. Therefore, if you have a spray dryer for a fine product, we do not offer much hope for making a coarse-product dryer out of it. Several attempts have been made to produce coarse product in small chambers, and they have been rather futile.

Anonymous: From what you say, that the hold-up time in the spray dryer is determined by the particle and the time it takes to dry that, is it possible to build a small unit for a pilot plant research, or do you have to have a large-scale model always?

W. R. Marshall: I believe that the desirable situation is to be able to design from data obtained on the largest possible unit. If one tries to get design data on too small a unit the attempt may fail altogether, or the resulting design may be unreliable.

Small spray dryers usually limit the handling equipment. In other words, it may be necessary to use dilute liquids to prevent plugging of feed lines and atomizers. In large-scale tests the conditions more nearly approach plant conditions, usually with much better operation. It may happen that a small-scale spray dryer test will fail completely when success would have been achieved in a large spray dryer.

Design data have been obtained in a 5-ft. dryer with reasonable success. However, that brings up the question, what about designing for large particles? It cannot be done in a dryer of this size.

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ENGINEERING ASPECTS OF SEPARATION OF GASES

FRACTIONAL PERMEATION THROUGH MEMBRANES

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Many nonporous organic membranes show widely varying permeabilities toward different gases. This property can be made the basis of a process for the separation of appropriate gas mixtures. The theory has been developed for the fractionation of a binary gas mixture, and the theory has been checked in a small, laboratory, single-stage unit operating on air at five atmospheres. Permeability coefficients toward a number of gases have been determined for a variety of commercial packaging films. Of the films tested, ethyl cellulose seems to be most suitable for the fractionation of air, and polystyrene most suitable for the separation of helium and hydrogen from gas mixtures.

Since the films are not perfectly selective, in general it is necessary to employ a multistage, recycle system to obtain gases of high purity, but the number of stages can be small in favorable cases. Application of the theory indicates that processes of potential commercial interest can be set up for the separation of oxygen from air, of helium from natural gas, and of hydrogen from such gases as coke-oven gas, tail-gas from hydrogenation processes, and petroleum refinery gases.

A NUMBER of organic membranes have the property of showing a much higher permeability toward some gases than toward others. For the occurrence of the phenomenon, it is necessary that the membrane be unbroken and nonporous, a requirement which distinguishes this process from those which depend on the operation of Graham's law in porous septa (3). Use of natural rubber membranes to achieve the separation of oxygen from air was suggested many years ago (4), but apparently the practical possibility of this and other gas fractionations has never been seriously investigated. This may be a result, in part, of the fact that until the development of modern plastics, only a limited number of materials was available in the form of thin films; of these, only rubber had the necessary properties of reasonable selectivity between gases and of sufficient permeability to permit significant quantities of gas to permeate under reasonable pressure gradients. The range of plastic

film compositions which is now available has greatly extended the choice in these directions, and it appears likely that some of the commercially available films are sufficiently selective, permeable, and stable to permit the development of economically feasible processes for the separation of appropriate gas mixtures.

Calculation of Enrichment in Single Stage

The theory for the fractionation of a binary gas mixture in a single stage of permeation has been previously developed (9). Differential rates of permeation of each component, *A* and *B*, through the film area *dS* obey Fick's law:

$$-dn_A = Q_A dS \left(P \frac{n_A}{n_A + n_B} - p \frac{n'_A}{n'_A + n'_B} \right) \quad (1)$$

$$-dn_B = Q_B dS \left(P \frac{n_B}{n_A + n_B} - p \frac{n'_B}{n'_A + n'_B} \right) \quad (2)$$

P is the high pressure, *p* the low pressure, *n_A* and *n_B* the volumes of *A* and *B*, respectively, flowing per unit time on the high pressure side, *n'_A* and *n'_B* the corresponding volumes flowing on the low pressure side, and *Q_A* and *Q_B* the permeability coefficients of *A* and *B* for the particular film.

The solution of Equations (1) and (2) depends on the nature of the gas flow on both sides of the film. It seems probable that any commercial application of a permeation process will require conditions of laminar, rather than turbulent, flow. The actual permeabilities of even thin films are quite low; therefore, in order that a significant fraction of the compressed gas which enters the stage should permeate through the film during the stage, relatively low flow rates would have to be employed. If laminar flow occurs on both sides of the film, and if the gas composition at any point on the low pressure side is given by the relative rates of permeation of the individual constituents at that point, then Equations (1) and (2) become

$$Q_A dS \left(P \frac{n_A}{n_A + n_B} - p \frac{dn_A}{dn_A + dn_B} \right) \quad (3)$$

$$Q_B dS \left(P \frac{n_B}{n_A + n_B} - p \frac{dn_B}{dn_A + dn_B} \right) \quad (4)$$

The solution of these equations has been shown (9) to be

$$\ln \frac{n_B^f}{n_B^o} = R \ln \frac{t^o - B/A}{t^o - B/A} + U \ln \frac{t^o - a + C}{t^o - a + C} + T \ln \frac{t^o - C}{t^o - C} \quad (5)$$

where the superscripts o and f refer to compressed gas entering and leaving the stage, respectively, and

$$a = Q_A/Q_B$$

$$A = [(1-a)p/P + a]/2$$

$$C = -[(1-a)p/P - 1]/2$$

$$B = -AC + a/2$$

$$R = 1/(2A - 1)$$

$$U = [a(A - 1) + C]/(2A - 1)$$

$$T = 1/(1 - A - B/C)$$

$$I = -Ai + [A^2i^2 + 2Bi + C^2]^{1/2},$$

$$i = n_A/n_B$$

For a given system, a , A , B , C , R , U , and T are constants and t^o is determined by the composition of the gas entering the stage. A value is assumed for the composition (n_A^f/n_B^f) of the high pressure, nonpermeated gas leaving the stage; this determines t^o . Equation (5) then permits the value of n_B^f to be calculated for any given value of n_B^o . The composition of the permeated gas is obtained from the values of n_A^o , n_A^f , n_B^o , and n_B^f ; the mole fraction of A in the permeated gas (y) is given by

$$y = (n_A^o - n_A^f)/(n_A^o - n_A^f + n_B^o - n_B^f), \quad (6)$$

and the fraction of the compressed gas entering the stage which permeates through the film during the stage (a) is given by

$$a = (n_A^o - n_A^f + n_B^o - n_B^f)/(n_A^o + n_B^o) \quad (7)$$

For a given set of conditions and a given feed, there is a particular value of y for each value of a ; curves showing this relationship for the fractionation of air are presented below.

The solution of Equations (3) and (4) is especially simple for the special case that the flow rate on the high pressure side is so great that $n_B^f = n_B^o$. Let the mole fraction of A in the permeated gas

$$\left(\frac{dn_A}{dn_A + dn_B} \right)$$

be y (a constant value) and the mole fraction of A on the high pressure side

$$\left(\frac{n_A}{n_A + n_B} \right)$$

be x (also constant). Dividing Equation (3) by Equation (4), there results

$$\frac{y}{1-y} = a \frac{Px - py}{P(1-x) - p(1-y)} \quad (8)$$

where $a = Q_A/Q_B$. For a given value of x , this quadratic equation may be solved for y .

For economic considerations it is necessary to know not only the enrichment obtained per stage, but also the area of film required to produce a given output of permeated gas. The exact solution is complicated, the area, S , being determined by the following:

$$S = \frac{n_B^o}{Q_B} \int_0^{i^o} \frac{\text{anti} \ln \left(\frac{n_B(i)}{n_B^o} \right) di}{[f(i) - 1] \left[P \frac{1}{i+1} - p \frac{1}{f(i)+1} \right]} \quad (9)$$

where

$$i = n_A/n_B$$

and

$$f(i) = Ai - C + [A^2i^2 + 2Bi + C^2]^{1/2}.$$

An estimate of S sufficiently good for preliminary engineering design can, however, be obtained in a simpler manner. The differential element of gas which permeates the film just at the inlet has a mole fraction of A , which

would be necessary to provide a permeation rate of Δn_A from a gas composition described by x^o on the high pressure side to a gas composition described by y^o on the low pressure side. By Equation (3),

$$S^o = \frac{\Delta n_A}{Q_A(Px^o - py^o)} \quad (10)$$

An analogous area, S' , can be calculated by

$$S' = \frac{\Delta n_A}{Q_A(Px^f - py^f)} \quad (11)$$

In most cases S^o and S' differ by less than a factor of two. The true area, S , required to obtain the flow Δn_A in the stage lies between the limits of S^o and S' , and it may be approximated, with a maximum error of perhaps 20%, by an arithmetic average of S^o and S' . This accuracy is probably sufficient for preliminary engineering estimates.

Film Permeabilities Toward Various Gases

A laboratory permeability study of a number of commercial film compositions has been made, and the results have been partially reported elsewhere (9). Of the films investigated, ethyl cellulose seems to be most generally suitable for the fractionation of air, and polystyrene seems to be outstanding for the separation of helium and hydrogen from gas mixtures. The permeability coefficients at 30°C. toward a number of gases for 0.001 in. ethyl cellulose and 0.001 in. polystyrene films are summarized in Table 1. From these data values may be calculated of the separation factors, $\alpha_{A/B}$, for any two gases A and B ; $\alpha_{A/B}$ is defined as Q_A/Q_B . For ethyl cellulose, for example, $\alpha_{O_2/N_2} = 3.4$, $\alpha_{H_2/CH_4} = 4.8$, and $\alpha_{N_2/CH_4} = 6.5$. For polystyrene $\alpha_{O_2/N_2} = 3.0$, $\alpha_{H_2/CH_4} = 15$, and $\alpha_{N_2/CH_4} = 20$. These separation factors are of primary importance in determining the power required for the fractionation of a binary gas mixture.

Data of Table 1 were obtained in low pressure laboratory experiments in which the gas permeated from atmospheric pressure into a vacuum. Verification of the results for a single stage has been made also on a laboratory

scale using compressed air at 5 atm. abs. on the high pressure side and atmospheric pressure on the low pressure side. Figure 1 shows the fractionation of air possible by a single-stage permeation process at a high pressure of 8 atm. or 5 atm. using a film with $\alpha_{O_2/N_2} = 3.4$. The curves indicate the theoretical oxygen content of the permeated air as a function of the fraction of inlet air which permeates during the stage; they were calculated by the use of Equation (5). The circles are experimental points obtained with a 5-atm. laboratory unit which employed a total area of about one square foot of 0.001 in. ethyl cellulose. The agreement with the theoretical curve is within experimental error. A second 5-atm. unit of similar capacity, operated under conditions of high gas flow on the high pressure side, has been producing 39-40% oxygen continuously for a period of more than five months; no change in its behavior was observed during that time. It is important for practical considerations that the film should be stable in this way, since frequent interruption of a commercial unit to replace film would be annoying and costly.

Application to Separation of Commercial Gas Mixtures

Since the films which are likely to be used in a permeation process are not perfectly selective, it will, in general, be necessary to employ a multistage, recycle system in order to obtain gases of high purity from a given gas mixture. The general principles of such multistage arrangements have been discussed by Benedict (1) and Cohen (2), among others. For a three-stage permeation process, for example, the simplified flow diagram would resemble that in Figure 2. The nonpermeated lean gas from each stage (except the first), which is still at high pressure, is sent back to the entrance of the previous stage. An interstage compressor is required between each pair of stages. Because of the cost of compressors, it will probably not be economical to have a stripping section. In the case of air fractionation, there would be no point in a stripping section since the feed gas is free and available in unlimited quantities; for other gases where the feed is limited and the desired component must be rather completely recovered, it will probably be least expensive to strip the component almost completely in the first stage, at the expense of the enrichment achieved in that stage, so that the amount of the component lost in the lean gas can be neglected.

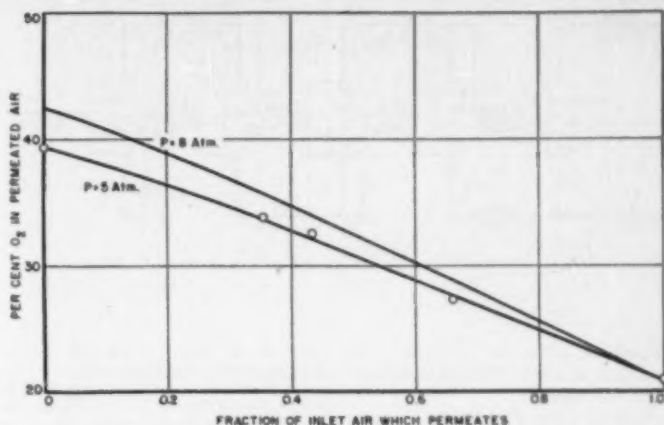


Fig. 1. Single-stage fractionation of air.

$$\alpha = Q_{O_2}/Q_{N_2} = 3.4.$$

Table 1.- Permeability Coefficients for Ethyl Cellulose and Polystyrene Films

| Gas | Permeability coefficient, Q (10^{-5} cu.ft./ (min.)(sq.ft.)(atm.)) | |
|--------------------------------|--|-------------|
| | Ethyl cellulose | Polystyrene |
| H ₂ | 1.94 | 1.5 |
| O ₂ | 6.6 | 4.4 |
| He | 21 | 24 |
| N ₂ | 29 | 33 |
| A | 4.5 | 3.2 |
| CO ₂ | 28 | 13.7 |
| CO | 2.1 | 1.9 |
| CH ₄ | 4.4 | 1.6 |
| C ₂ H ₆ | - | 1.5 |
| C ₃ H ₈ | 7.0 | 1.6 |
| C ₄ H ₁₀ | - | 3.0 |
| C ₅ H ₁₂ | - | 1.6 |

The treatment of the lean gas from the first stage depends on the individual case. In the case of oxygen separation from air, the compressed lean air from the first stage still contains appreciable amounts of oxygen, and it may conveniently be used with an appropriate fuel to drive a gas turbine for compressing

the air. In cases where the feed gas to the first stage is furnished under compression, which would be true for various plant gases, it is doubtful that the compressional energy of the first stage lean gas would be recovered. Where the feed gas has to be compressed, however, the lean gas would probably be passed

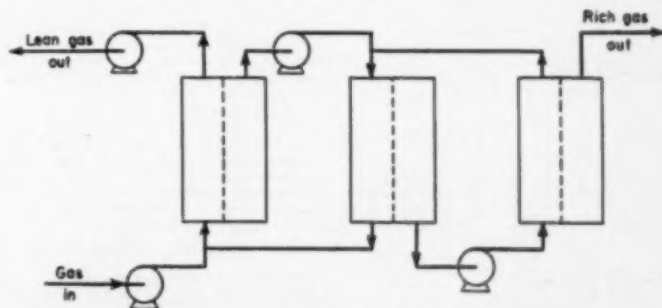


Fig. 2. Simplified flow diagram for a three-stage permeation process.

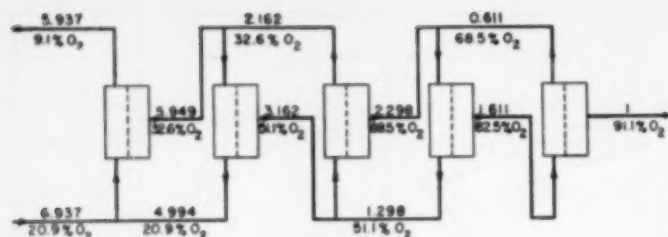


Fig. 3. Material flow diagram for multistage enrichment of air.

through an expansion engine in order to recover as much of the compressional energy as possible.

In the discussion which follows, several examples of gas separations are considered. The treatment of each example is not intended to be exhaustive. For one thing, each gas mixture is considered equivalent to a binary system; for another, no detailed investigation of process variables has been made to determine the economically optimum set of operating conditions. (As a matter of fact, it will be impossible to make the latter determination until some evaluation can be made of the cost of large film cases.) Results, therefore, are to be taken only as preliminary, qualitative indications of possible processes based on gas permeation through films.

Example 1. Single-stage Enrichment of Air in Oxygen. A 0.001-in. ethyl cellulose film is to be used, for which $\alpha = Q_{O_2}/Q_{N_2} = 3.4$. The high pressure is arbitrarily taken as 8 atm. (that being a convenient upper limit for a two-stage turbo-compressor) and the low pressure as 1 atm. abs. The variation in composition of the permeated air as a function of fraction of inlet air which permeates is shown in Figure 1. Suppose air containing 32.6% O_2 is desired. Then 50.2% of the air which enters the stage must permeate during the stage. The corresponding flow diagram is shown in Figure A.

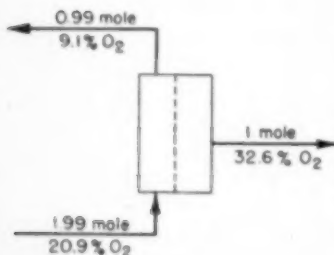


Fig. A.

Per mole of 32.6% O_2 out, 1.99 mole of normal air has to be compressed to 8 atm., and 0.99 mole of air containing 9.1% O_2 are to be decompressed. (Although the most efficient utilization of the lean air is probably for combusting fuel in driving a gas turbine, for simplicity an expansion engine will be considered here.) Assuming an adiabatic efficiency of 80% for the two-stage compression to 8 atm. and 75% for the expansion, the net work required is 3.4 kw.hr./M. cu.ft. of air containing 32.6% O_2 . This may be compared with the corresponding work required by a Linde process. A modern Linde oxygen plant has a power requirement of about 15 kw.hr./M. cu.ft. of air containing 95% O_2 . Since 0.158 volumes of (air containing) 95% O_2 can be diluted with normal air to make 1 volume of 32.6% O_2 , the work by a Linde process is 2.4 kw.hr./M cu.ft. of 32.6% O_2 . This is lower than the work in the permeation process by 30%, but certainly of the same order of magnitude.

Because of the low absolute permeabilities of the films, large areas are, in general, required in order to have gas productions of commercial interest. To produce 2000 cu.ft./min. of 32.6% O_2 , for example, a total area of 11.5×10^6 sq.ft. of 0.001-in. ethyl cellulose would be required, or 6000 sq.ft./cu.ft.(min.). (This value was obtained by the approximation procedure indicated above; it was the average of 10.4×10^6 sq.ft. required for gas compositions corresponding to the inlet, and 12.5×10^6 sq.ft. required at the outlet.) Some conception of the space needed to house this film area can be obtained in the following way: It is possible to design a multilayer film sandwich so that at least 100 films can be packed per linear foot. This corresponds to an area of 100 sq.ft. of film which can be packed in a volume of 1 cu.ft. On the assumption that this packing density can be maintained in a large film pack, the required area of 11.5×10^6 sq.ft. could be encompassed in a volume of 115,000 cu.ft., or a space 95 ft. \times 50 ft. \times 25 ft. In practice, because of the fact that the film sandwiches would have to be enclosed in pressure casings, it is likely that the total volume required to house the film would be about double this figure. It is convenient, however, to maintain this simple estimation of space requirements for rapid, order-of-magnitude calculations.

The weight of ethyl cellulose required to make 11.5×10^6 sq.ft. of 0.001 in. film is about 70,000 lb. A single compressor is required; for the assumed production of 2000 cu.ft./min. of 32.6% O_2 , its capacity would be 4000 cu.ft./min.

Example 2. Multistage Enrichment of Air. In case O_2 of 90-95% purity is required, a multistage system must be used. As in Example 1, ethyl cellulose, with $\alpha = 3.4$, will be considered, and again the arbitrary pressure of 8 atm. will be used as the high pressure P . The lean air from the first stage is assumed to contain 9.1% O_2 (see Example 1); this choice, plus the condition that the lean air from any higher stage must have the same composition as the air entering the next lower stage, is sufficient to determine uniquely the entire cascade. The material flow diagram arrived at is shown in Figure 3. The upper figure for each gas stream is the number of moles of gas flowing in the stream per mole of air containing 91.1% O_2 produced by the fifth stage; the lower figure gives the oxygen content of the particular stream. Five stages are required to produce oxygen in the desired concentration range.

In Table 2 are summarized the conditions of each of the five stages for a plant producing 2000 cu.ft./min. of 91% O_2 . The data include the fraction of air entering a stage which permeates during that stage, the composition of the permeated air, the output of each stage, the compressor requirements, and the area of film needed.

Per mole of product air, a total of 20 moles of air has to be compressed to 8 atm., of which 5.9 moles (lean air from the first stage) is to be decompressed. Again assuming an adiabatic efficiency of 80% for compression and 75% for expansion, the net work may be calculated to be 40 kw.hr./M cu.ft. of 91% O_2 . This is between two and three times the work required in a Linde plant.

The total film area required is 133×10^6 sq.ft., or 67,000 sq.ft./cu.ft.(min.). On the basis of the packing assumption made in Example 1, namely, that 100 sq.ft. of film can be contained in 1 cu.ft., this area would require a total volume of 133×10^6 cu.ft., which is roughly equivalent to a building 220 ft. \times 200 ft. \times 30 ft. It should be kept in mind that this type of size estimation is only a crude approximation.

Example 3. Separation of Helium from Natural Gas. Assume natural gas containing 1.00% He is available at a pipe-line pressure of 15 atm. A recovery of at least 90% He is desired. The major gas constituents which have to be considered are CH_4 , He, and N_2 . Inspection of Table 1 shows that for polystyrene films the separation factor of He vs. both N_2 and CH_4 is about 15; i.e., $Q_{He}/Q_{N_2} = Q_{He}/Q_{CH_4} = \alpha = 15$. For the purpose of this separation, therefore, the natural gas may be considered as a mixture of 1.00% He and 99.00% CH_4 . On the assumption that the lean gas from the first stage contains 0.13% He, a six-stage permeation process may be set up to produce 99.4% He with a recovery of 90.5% of the helium. The material flow diagram is indicated in Figure 4. A high pressure of 15 atm. is used throughout. Gas is furnished to the first stage already under compression, and the lean gas from the first stage is returned to the pipe line without further compression. Only five interstage compressors are required; these serve to compress to 15 atm. a total of 56 moles of gas per mole of 99.4% He produced. Assuming three-stage adiabatic compression with an efficiency of 80%, the total work of compression is, therefore, 182 kw.hr./M cu.ft. of 99.4% He. Com-

pression of the product helium to 2000 lb./sq.in. would add only about 7 kw.hr./M cu.ft. The total is well under the rough figure of 740 kw.hr./M cu.ft. of 98% He estimated for the Bureau of Mines low temperature process (5). The reason for the lower power requirement in the permeation process is that the bulk of the natural gas treated is returned still under pressure as lean gas from the first stage, while in the low temperature separation all of the natural gas is expanded from pipe-line pressure to about 70 lb./sq.in. (6) in order to obtain cooling, and it has to be subsequently recompressed to pipe-line pressure.

The total production of helium in the United States during 1946 was 63.4×10^6 cu.ft. (8), which corresponds roughly to 120 std.cu.ft./min. The last three columns of Table 3 were calculated for a plant of this capacity, i.e., one which could furnish the entire production of the United States. A total film area of 35.8×10^6 sq.ft. would be required, of which the greatest part is needed for the first stage. Using the order-of-magnitude packing density just mentioned, this area could be accommodated in a volume of 3.6×10^6 cu.ft., corresponding to a building 100 ft. \times 100 ft. \times 36 ft.

Example 4. Separation of Hydrogen from Hydrogenation Tail-gas. Assume H_2 of at least 98% purity is to be separated from the tail-gas of a high pressure hydrogenation process; almost complete recovery of H_2 is desired. The gas contains 60% H_2 , 21% CH_4 , 7% C_2H_6 , 5% C_3H_8 , 4% CO , and 3% N_2 . (This is the approximate composition of coal hydrogenation tail-gas after water scrubbing (7). Reference to Table 1 shows that if a polystyrene film is used in the permeation process, the tail-gas may, to a good approximation, be considered as a binary mixture of 60% H_2 and 40% CH_4 , for which the separation factor $\alpha = Q_{H_2}/Q_{CH_4} = 20$. The gas may be considered to be furnished to the first permeation stage at a pressure of 15 atm.; higher stages are also run at this pressure.

As in the case of helium separation, it is preferable to effect almost complete stripping of the desired gas in the first stage. A choice of lean gas composition of 38% H_2 (from the first stage) corresponds to a 98% recovery of H_2 from the entering gas. As indicated in Figure 5 and Table 4, this choice permits setting up a two-stage separation process which produces 85.9% H_2 in the first stage and 98.4% H_2 in the second. Data of Table 4 are calculated for a plant of 2000 cu.ft./min. capacity. Only a single interstage compressor is needed, of capacity 3000 cu.ft./min. Assuming three-stage adiabatic compression with an efficiency of 80%, the compressional work amounts to 4.5 kw.hr./M cu.ft. of 98.4% H_2 .

The total film area required is 4.9×10^6 sq.ft., or 2500 sq.ft./cu.ft. (min.). Although the first stage output is only 50% greater than that of the second stage, it requires seven times as much film area. This is a result of the large difference of permeability coefficients between H_2 and CH_4 . As a matter of fact, this difference, plus the large change in composition of the high pressure gas as it passes through the first stage, causes the approximation procedure used to calculate area to be relatively poor for the first stage. S^0 (Eq. (10) above) in this case is 1.1×10^6 sq.ft., and S^1 (Eq. (11)) is 9.3×10^6 sq.ft., with

Table 2.—Multistage Enrichment of Air

| Stage No. | Fraction of in-gas permeating | % O_2 in permeated gas | Stage output, cu.ft./min. | Compressor capacity, cu.ft./min. | Film area, 10^6 sq.ft. |
|-----------|-------------------------------|--------------------------|---------------------------|----------------------------------|--------------------------|
| 1 | 0.502 | 32.6 | 11,990 | 13,970 | 68.7 |
| 2 | .388 | 51.1 | 6,320 | 11,990 | 30.3 |
| 3 | .515 | 68.5 | 4,500 | 6,320 | 18.1 |
| 4 | .554 | 82.5 | 3,220 | 4,600 | 10.3 |
| 5 | .621 | 91.1 | 2,000 | 3,220 | 5.6 |
| | | | | 40,010 | 132.0 |

Table 3.—Separation of Helium From Natural Gas

| Stage No. | Fraction of in-gas permeating | % He in permeated gas | Stage output, cu.ft./min. | Compressor capacity, cu.ft./min. | Film area, 10^6 sq. ft. |
|-----------|-------------------------------|-----------------------|---------------------------|----------------------------------|---------------------------|
| 1 | 0.267 | 3.4 | 4,930 | 0 | 21.1 |
| 2 | .185 | 13.9 | 1,100 | 4,930 | 12. |
| 3 | .273 | 41.8 | 370 | 1,100 | 2.3 |
| 4 | .420 | 80.3 | 180 | 370 | .4 |
| 5 | .707 | 96.3 | 140 | 180 | .07 |
| 6 | .836 | 99.4 | 120 | 140 | .06 |
| | | | | 6,720 | 35.8 |

Table 4.—Separation of Hydrogen From Hydrogenation Tail-Gas

| Stage No. | Fraction of in-gas permeating | % H_2 in permeated gas | Stage output, cu.ft./min. | Compressor capacity, cu.ft./min. | Film area, 10^6 sq.ft. |
|-----------|-------------------------------|--------------------------|---------------------------|----------------------------------|--------------------------|
| 1 | 0.685 | 85.9 | 2960 | 0 | 4.3 |
| 2 | .675 | 98.4 | 2000 | 2960 | .6 |
| | | | | 2960 | 4.9 |

Table 5.—Enrichment of Refinery Gas

| Stage No. | Fraction of in-gas permeating | % H_2 in permeated gas | Stage output, cu.ft./min. | Compressor capacity, cu.ft./min. | Film area, 10^6 sq.ft. |
|-----------|-------------------------------|--------------------------|---------------------------|----------------------------------|--------------------------|
| 1 | 0.124 | 47.1 | 407 | 0 | 0.96 |
| 2 | .492 | 85.4 | 200 | 407 | .24 |
| | | | | 407 | 1.20 |

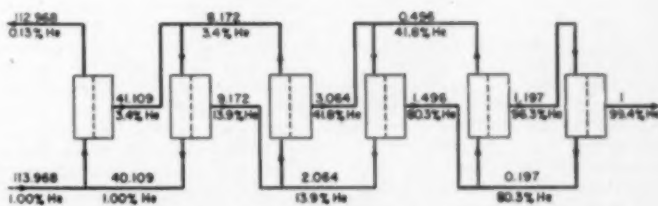


Fig. 4. Separation of helium from natural gas.

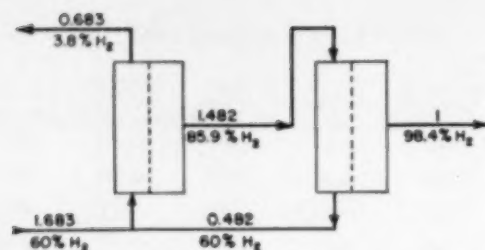


Fig. 5. Separation of hydrogen from hydrogenation tail-gas.

an average value of 5.2×10^6 sq. ft. Even in this extreme case the average value turns out to be reasonably close to the exact value, 4.3×10^6 sq. ft., determined by the use of Equation (9). If the packing density of 100 sq. ft. of film/cu. ft. of volume can be maintained, the total volume required to contain the area of 4.9×10^6 sq. ft. would be about 5×10^6 cu. ft., equivalent to a building 50 ft. \times 50 ft. \times 20 ft.

It may be noted in connection with this example that if an enrichment of the hydrogen from 60% to 86% is sufficient, a single-stage fractionation may be employed in which 98% of the total hydrogen is recovered in the rich stream, and in which no compression need be furnished unless the rich gas must be available under pressure. The plant size would, however, be almost as large as that for the two-stage process.

Example 5. Enrichment of Refinery Gas. Petroleum refinery gas containing 10% H_2 and 90% N_2 , CH_4 , C_2H_6 , C_3H_8 , C_4H_{10} , and C_5H_{12} is available at 15 atm. pressure. A recovery of at least 50% H_2 is required, and the enriched gas must contain at least 80% H_2 for use in a hydrogenation process.

With the use of polystyrene films, a two-stage process may be set up as indicated in Table 5. A total of 58% hydrogen is recovered, and the product gas contains 85% H_2 . Table 5 has been calculated on the assumption that an output of 200 cu. ft./min. (288,000 cu. ft./day) is desired. Unless the enriched gas has to be recompressed, only one interstage compressor, of capacity 410 cu. ft./min., is required. The corresponding work is 6.3 kw./hr./M cu. ft. A film area of 6000 sq. ft./cu. ft. (min.) of gas is needed; the total area of 1.2×10^6 sq. ft. requires (by the previous approximation) a volume of 1.2×10^6 cu. ft., equivalent to a building 30 ft. \times 20 ft. \times 20 ft.

Summary

The preceding examples are typical gas separations which can be effected with the use of films already studied. For appropriate gas mixtures, the power requirements of a permeation process are of the same order of magnitude as those for low temperature separations. Because of low film permeabilities, relatively large plants will, in general, be required. It is important to note, however, that the total film area (i.e., plant size) needed is approximately inversely proportional to the pressure used on the high pressure side. Within limits, therefore, doubling the pressure decreases the plant size roughly by a factor of

two;† this is done, of course, at the expense of increasing the compressional power requirement. Determination of the economically optimum pressure would depend on a detailed balancing of the cost of large-scale film packs and the cost of compression.

On the basis of the work done thus far, it appears that the production of pure oxygen by a permeation process does not seem to be competitive with the Linde process because of both high power requirements and large plant size. Production of slightly enriched air by a single-stage permeation process is much closer to being of practical interest and should be looked at more closely. Helium separation from natural gas appears promising from the viewpoint of power consumption, and the relatively small total production of helium means that the plant need not be inordinately large. The enrichment of hydrogen-containing gases is also interesting because of the large separation factors which can be obtained with polystyrene films.

Any large-scale application of permeation processes for gas separation must be predicated on the development of cheap film cases. The films must, of course, be supported in order to withstand superatmospheric pressure, and the engineering problems associated with doing this economically have not yet been worked out. Designs for a prototype of one possible film case have been made, but until the model has been constructed and tested, it will not be possible to evaluate the cost of plant construction.

It should be kept in mind that all the work carried out thus far has been with common, commercially available packaging films. In view of the fact that there seems to be no correlation between the chemical nature of a film and its separative properties, it is almost certain that the optimum film has not been found for any given gas separation. It would be highly desirable to make a systematic study of a much larger range of

† Actually, by a factor greater than two, since the enrichment is improved by an increase in pressure (see Fig. 1).

film compositions, since major improvements may be found in film selectivities and permeabilities which would vitally affect the feasibility of certain gas separations.

Notation

Q = permeability coefficient, cu. ft./ (min.) (sq. ft.) (atm.)

S = film area, sq. ft.

P = total pressure on high pressure side, atm.

p = total pressure on low pressure side, atm.

n = flow rate on high pressure side, cu. ft./min.

n' = flow rate on low pressure side, cu. ft./min.

y = mole fraction of more permeable constituent in permeated gas

x = mole fraction of more permeable constituent in non-permeated gas

a = fraction of inlet gas which permeates during stage

α = ratio of permeability coefficients of constituents in a binary mixture

SUPERSCRIPTS:

\circ = inlet on high pressure side

f = outlet on high pressure side

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(Presented at Forty-second Annual Meeting, Pittsburgh, Pa.)

RADIOISOTOPES

L. B. EMLET

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RADIOACTIVE isotopes have been available in limited supply for a number of years. Prior to 1943, however, these materials were produced mainly by the bombardment of target nuclei by particles accelerated in a cyclotron. With the advent of the nuclear reactor (Dec. 2, 1942), a potentially larger and cheaper source of radioisotopes became available. The security problems surrounding the Atomic Energy Project during the war years limited the distribution of radioactive materials. In August, 1946, however, the radioisotopes prepared at the Oak Ridge National Laboratory were offered to all qualified persons in the country. Later (September, 1947) the distribution program was expanded to make certain of these radioactive materials available to research institutions in foreign countries.

Radioisotopes are excited atoms which are attempting to reach a stable state by emitting energy and/or nuclear particles. Regardless of whether the radioisotope is produced in a cyclotron or in a pile, the process involves upsetting the balance of particles within the nucleus of the atom.

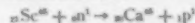
Figure 1 illustrates a graphite reactor similar to the one located at Oak Ridge National Laboratory. The pile proper—that is, the uranium and graphite—is enclosed inside a heavy shield made of concrete. The concrete structure of the Oak Ridge pile is 47 ft. long, 38 ft. wide, and approximately 35 ft. high. The wall thickness is about 7 ft. Inside this concrete shield is a stack of graphite blocks. It is into these holes that the uranium fuel rods are placed. Other holes enter the graphite-uranium structure from the top and sides. These channels provide space for the control rods, research experiments and for the exposure of target materials used in preparing some of the radioisotopes. Large quantities of air are pulled through the pile to carry away the heat generated by the nuclear reactions.

AUTHOR'S NOTE: Since the release of this article, it has been discovered at the Oak Ridge National Laboratory that ^{135}Te probably does not exist. This fact, however, does not influence the validity of the effect which this radioisotope was used to illustrate.

Figure 2 illustrates the nuclear reactions which take place in the pile:

1. A slow neutron, wandering around in space, strikes one of the atoms of ^{235}U , which is present in the normal fuel rods to the extent of 0.7%. This ^{235}U atom splits forming two fission fragments (two elements lower in the atomic table), energy, and two or three new neutrons.
2. These newly born neutrons lose energy by wandering through the graphite blocks and finally collide with more ^{235}U atoms, which also undergo fission. In a fraction of a second there are millions of neutrons within every cubic centimeter of the pile. The Oak Ridge pile has a neutron flux of about 5×10^{13} neutrons/(sq.cm.) (sec.).
3. Some of the neutrons strike nuclei of ^{235}U atoms and are absorbed to form ^{236}U , which decays by beta emission to form ^{236}Np . The ^{236}Np decays by emitting an electron from the nucleus (beta particle) to form ^{236}Pu .
4. Other neutrons are absorbed by the nuclei of various target elements, which have been placed in the exposure channels, thereby producing radioisotopes.
 - a. The first case illustrated provides for the production of carrier-free radioactive iodine. The ^{135}Te nucleus absorbs a neutron to form ^{136}Te , which decays by beta emission to ^{136}I . The ^{136}I is then separated and purified. Any reaction which produces a carrier-free radioisotope is particularly important, since most customers want the active element undiluted with its stable isotopes.

- b. In the second case the neutron enters the nucleus of a ^{197}Au atom to form radiogold (^{198}Au). The ^{197}Au decays by beta emission to stable ^{198}Hg .
- c. In the third case illustrated, the neutron enters the nucleus of a scandium atom and drives out a proton, thus changing the scandium into radiocalcium.



This reaction also produces a carrier-free radioisotope; i.e., radiocalcium that is not diluted with stable calcium.

- d. Another source of many radioisotopes is the fission fragments resulting from the splitting of the ^{235}U nucleus. In the normal fission spectrum, about 34 elements are found with several different radioisotopes for each. They run from Zn (At No. 30) to Gd (At No. 64). When a certain quantity of ^{235}U undergoes fission, it produces roughly an equal weight of fission products. Under normal operating conditions with the fission products at equilibrium, the pile contains radiation equivalent to that from about 30 tons of radium.

The production rate of radioisotopes or the rate of activation of target materials placed in the pile is dependent upon several different factors. They are:

1. Available neutron flux or the number of neutrons/(sq.cm.) (sec.). In the Oak Ridge pile this is about 5×10^{13} neutrons/(sq.cm.) (sec.).
2. Energy of the neutron.

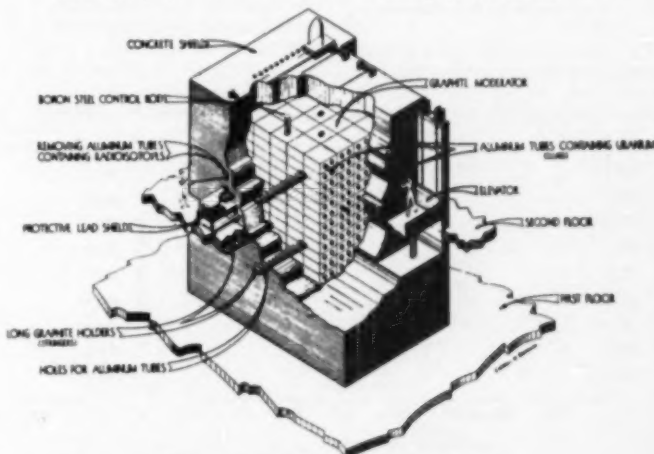


Fig. 1. Nuclear reactor—uranium "pile."

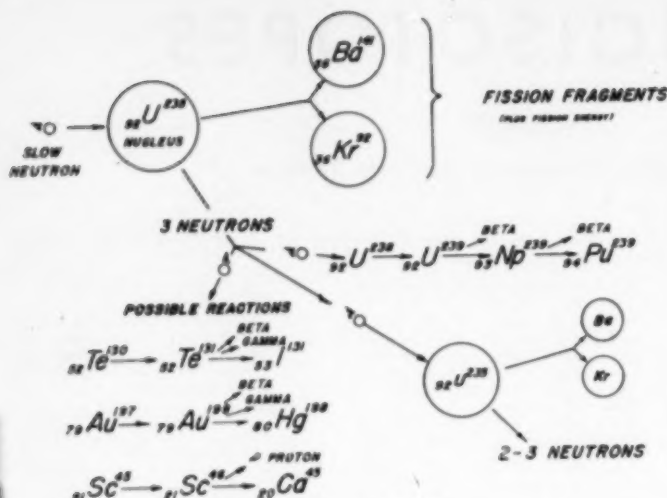


Fig. 2. Neutron reactions.

TABLE 1.

| Target Element | % Abundance | Activity | T% | Activation of Atom of Normal Element (10 ⁻⁴¹ sq.cm.) |
|---------------------------------|-------------|---------------------------------|---------|---|
| ¹¹ Na ²³ | 100.0 | ¹¹ Na ²⁴ | 14.8 h | .4 |
| ²⁰ Ca ⁴⁰ | 96.9 | ²⁰ Ca ⁴⁵ | 8.5 d | .0003 |
| ⁴⁰ Ca ⁴⁴ | 2.0 | ⁴⁰ Ca ⁴⁷ | 180.0 d | .5 |
| ⁵⁰ Cr ⁵² | 100.0 | ⁵⁰ Cr ⁵³ | 85.0 d | 22.0 |
| ⁵⁹ Fe ⁵⁶ | 5.3 | ⁵⁹ Fe ⁶⁰ | 72.0 d | 1002 |
| ⁵⁹ Co ⁵⁹ | 100.0 | ⁵⁹ Co ⁶⁰ | 5.3 y | 33.5 |
| ⁶³ Tb ¹⁵⁹ | 100.0 | ⁶³ Tb ¹⁶⁰ | 72.0 d | 100.0 |

TABLE 2.

| Isotope | Half-Life | Wt./Curie gram | Micrograms |
|-------------------|-----------|----------------------|------------|
| ¹³¹ I | 8-day | 8 × 10 ⁻⁶ | 8 |
| ^{131m} I | 14.3-day | 2 × 10 ⁻⁶ | 2 |
| ⁵⁹ Fe | 45-day | 5 × 10 ⁻⁶ | 5 |
| ⁶⁰ Co | 5.3 year | 9 × 10 ⁻⁶ | 900 |
| ¹³⁷ Cs | 1100-year | 0.3 | |
| ²²⁶ Ra | 1580-year | 1.0 | |

- The cross section or affinity of the particular target element for absorbing neutrons.
- The half-life of the radioisotope being produced.

The first two factors are largely dependent upon the pile design; therefore, only the last two are considered. The cross section is generally defined as the

probability of the interaction between the target nuclei and the number of incident neutrons. This cross section is dependent upon many factors, such as energy of the neutrons, type of reaction (α , γ or n, p), thickness of target, etc. The cross section for any given reaction is normally expressed in

"barns," where a barn is 10⁻²⁸ sq.cm.

Table 1 illustrates a few cross sections for the common n, γ reaction.

The half-life of a radioisotope is a constant in the same way that Avogadro's number is accepted as a constant. No known force can lengthen or shorten the rate of decay of a radioactive isotope.

Figures 3 and 4 and Tables 3 and 4 illustrate the effect of cross section and the half-life on the rate of production of several different radioisotopes.

In one case, ⁶³Tb¹⁶⁰ with a cross section of 100 b (100 × 10⁻²⁸) is compared to ⁵⁹Fe⁶⁰ with a cross section of 0.002 b (0.002 × 10⁻²⁸). The half-life of each is 72 days. Although the two materials approach their saturation point at an equal rate, the quantity of ⁶³Tb¹⁶⁰, which can be produced, greatly exceeds the amount of ⁵⁹Fe⁶⁰ of target material.

In the second case, both Sc and Co have essentially the same cross section—about 22 b (22 × 10⁻²⁸)—but the half-life of Sc⁴⁶ is 85 days, while the Co⁶⁰ has a half-life of 5.3 years. At saturation each has the same number of disintegrating atoms per mole, although the Co contains 227 times as many unstable atoms as does the Sc and will be radiating long after the Sc⁴⁶ is depleted.

The basic equation for calculating the rate of formation of a radioisotope is given by:

$$C/\text{mole} = \frac{(N \times \sigma \times nv)}{(3.7 \times 10^{10})} (1 - e^{-\lambda t})$$

C = curies

N = number of atoms of target material exposed

σ = cross section in sq.cm.

nv = number of neutrons directed at target material/(sq.cm.) (sec.)

n = number of neutrons/cc.

v = velocity of neutrons in cm./sec.

3.7×10^{10} = disintegrations/sec. in curie of material

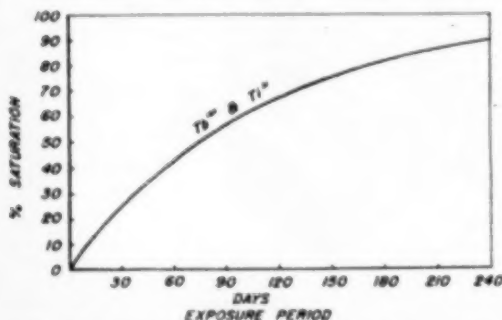


Fig. 3. Exposure period vs. % saturation Tb¹⁶⁰ and Ti⁴⁴.

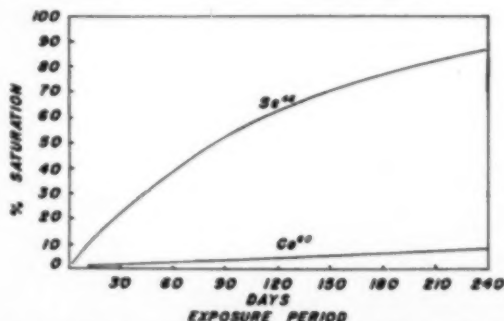


Fig. 4. Exposure period vs. % saturation Sc⁴⁶ and Co⁶⁰.

λ = decay constant = $0.693/T_{1/2}$
 $T_{1/2}$ = half-life of radioisotope
 t = time of exposure

By substituting numerical values:

$$C/\text{mole} = \frac{\left(\frac{\text{Avogadro's No.}}{6 \times 10^{23}} \right) \left(\frac{\text{Cross Section}}{\sigma \times 10^{-24}} \right) \left(\frac{\text{Neutron Flux}}{5 \times 10^{11}} \right) \left(\frac{\text{Decay Factor}}{1 - e^{-\lambda t}} \right)}{\left(\frac{\text{dis./sec.}}{3.7 \times 10^{10}} \right)}$$

Assume that one gram of cobalt metal is exposed at 66% maximum neutron flux for 60 days. The cross section for an n, γ reaction on cobalt is 22.5×10^{-24} sq.cm., while Co^{60} has a half-life of 5.3 years (1930 days).

$$C/\text{mole at Saturation} = \frac{(6 \times 10^{23})(22.5 \times 10^{-24})(.66 \times 5 \times 10^{11})}{(3.7 \times 10^{10})} = 120 \text{ C.}$$

$$\left(\frac{C/\text{mole}}{60 \text{ days}} \right) = 120 \times (1 - e^{-\lambda t})$$

$$\left(1 - e^{-\frac{0.693 t}{T_{1/2}}} \right) = 0.02$$

0.693 = natural logarithm of 2

$T_{1/2}$ = half-life in days

t = exposure period in days

e = base for natural logarithms

$$\left(\frac{C/\text{mole}}{60 \text{ days}} \right) = 120 \times 0.02 = 2.4 \text{ C/mole}$$

$$C/\text{g.} = \frac{2.4}{60} = 0.04 \text{ C or } 40 \text{ mc.}$$

The mass associated with a curie of a radioactive compound, particularly those of short half-life, is extremely small. By definition, one gram of Ra^{226} under-

goes 3.7×10^{10} dis./sec., which is called a curie. Table 2 lists the approximate mass/curie of some of the radioisotopes now being distributed.

In most radiochemical separation processes, one is confronted with two

major problems: (1) Radioactivity which requires remote control operation through thick shields, and (2) Extremely small quantities of product in comparatively large amounts of target

material. Some of the chemical processes are completed entirely behind 3 to 5 ft. of concrete and result in only a few micrograms of product, although several pounds (perhaps 100,000,000 times the weight of the radioisotope) of target material are handled.

The chemical separation and purification of radioactive compounds can be illustrated by the flow sheet (Fig. 5) for the preparation of radiophosphorus:

1. Four kg. of irradiated sulfur are melted out of the aluminum exposure can and placed in an autoclave containing 1 l. of 0.2N HNO_3 .
2. With the temperature at $120^\circ\text{--}130^\circ\text{C}$. and the pressure at 25 lb./sq.in., the mix is agitated and allowed to settle.
3. The sulfur is separated and discarded. The aqueous phase, containing the P^{32} as $\text{H}_2\text{P}^{32}\text{O}_4$, is filtered to remove the remaining sulfur.
4. The filtrate is adjusted to a 2.5 to 5% caustic with 33% NaOH . Contaminating materials—iron, nickel, chromium—precipitate as hydroxides and are removed by filtration. The P^{32}O_4 is not carried on the hydroxides at this high a pH. The filtrate is acidified with HNO_3 , and 200 mg. of lanthanum nitrate added.

5. The solution is then adjusted to a pH of 8 or 9 with NH_4OH . The precipitated lanthanum hydroxide carries the P^{32} as lanthanum phosphate.

6. After filtration, the precipitate is dissolved in 20 ml. of hot 6N HCl and diluted to 1 l.

7. The acid solution is passed through a 1×20 cm. Dowex No. 50 resin column to remove all cations.

8. The column effluent is evaporated to dryness at 90°C . and dried for 1 hr. (If large losses are to be avoided, carrier should be added just before evaporation, —25 micrograms/mc P^{32} .) The residue is dissolved in water, filtered, and analyzed.

9. The final solution of about 250 ml. normally contains about one curie of P^{32} (3 micrograms) and about 25 milligrams of carrier. Our selling price is \$110/mc., the batch being worth about \$1100.

10. Our specifications for P^{32} are:

| Spec. Item | Specification |
|----------------------------------|---------------------------------------|
| Preparation | Phosphate in weak HCl |
| Concentration, radioelement | >0.5 mc./ml. |
| Concentration, total element | 0.025 mg. P added/mc. P^{32} |
| Radiochemical purity | 99.9% |
| Total solids | <5 mg./ml. |
| N V M | <5 mg./ml. |
| Color and clarity | White, clear |
| pH | 1.0-5.0 |
| Prts. H ₂ S at 7.0 pH | <10 p.p.m. |
| Prts. at pH 7.0 | No precipitate in 24 hr. |
| Aluminum | <1 p.p.m. |

At the present time approximately 85 to 90% of all radioisotopes distributed have been used in biological or medical research. An increasing number of industrial laboratories, however, are now finding these radioactive materials of value in solving their problems.

Several of this country's large oil

| TABLE 3 | | | |
|-----------------------------|---------------------------------------|---------------------------------------|--|
| Terbium— mTb^{140} | | Titanium— mTi^{48} | |
| $T_{1/2} = 72$ days | | $T_{1/2} = 72$ days | |
| Cross section = 100b | | Cross section = 0.002b | |
| Exposure Period Days | Yield (millicuries/g. Normal Element) | Yield (millicuries/g. Normal Element) | |
| 30 | 1,290 | 0.082 | |
| 60 | 2,190 | 0.140 | |
| 90 | 2,920 | 0.190 | |
| 120 | 3,450 | 0.220 | |
| 150 | 3,860 | 0.250 | |
| 180 | 4,160 | 0.260 | |
| 210 | 4,420 | 0.280 | |
| 240 | 4,570 | 0.290 | |
| Saturation | 5,080 | 0.320 | |

Flux: $ne = 5 \times 10^{11}$

| TABLE 4 | | | |
|-----------------------------|---------------------------------------|---------------------------------------|--|
| Scandium— mSc^{46} | | Cobalt— mCo^{60} | |
| $T_{1/2} = 85$ days | | $T_{1/2} = 5.3$ years | |
| Cross section = 22.0b | | Cross section = 22.5b | |
| Exposure Period Days | Yield (millicuries/g. Normal Element) | Yield (millicuries/g. Normal Element) | |
| 80 | 850 | 33 | |
| 80 | 1,520 | 65 | |
| 90 | 2,050 | 99 | |
| 120 | 2,440 | 129 | |
| 150 | 2,760 | 160 | |
| 180 | 3,030 | 191 | |
| 210 | 3,230 | 222 | |
| 240 | 3,390 | 249 | |
| Saturation | 3,940 | 3080 | |

Flux: $ne = 5 \times 10^{11}$

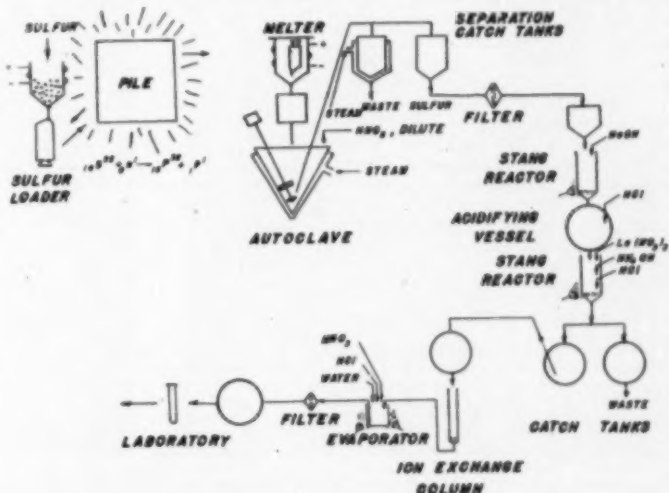


Fig. 5. P^{32} flow sheet.

companies are profiting by the use of radioactive materials. For example:

1. A rather common method employed to clean out the long oil lines is to force a "go-devil" or scraper through the line by hydraulic pressure. At various points along the line, ports or outlets are provided through which the cleanings are removed. Occasionally the "go-devil" becomes stuck in the line between two of the outlets. In the past it was necessary to "guess" the position of the scraper and dig up many sections of pipe until the "go-devil" was found. Some oil companies are now placing a small Co^{60} source on the scraper. If this scraper is stuck, a radiation detection instrument rapidly locates its exact position and eliminates digging up more sections of pipe than necessary.
2. Experiments have been run on the use of radioactive Ba^{133} as a means of distinguishing between two different grades of oil in a long pipeline. It is common practice to use the long underground pipelines to carry gasoline, fuel oil, etc. Density measurements have been used in the past to determine where the one fluid stopped and the other started. By introducing Ba^{133} at the interface of the two liquids, a G-M tube can now make this determination more precisely and with much less expenditure of time and effort.

Diffusion problems can easily be investigated by the use of radioactive isotopes. For example, a stainless steel block containing radio- Cr^{51} is clamped to a nonactive block and the two heated for a given period. After cooling, thin slices of the nonradioactive block will, upon examination, show the presence of Cr^{51} , indicating diffusion of metal atoms from one block into the other.

Many industries are faced with the problem of obtaining a completely homogeneous product by milling or agitation. Some process control tests used to determine the completeness of mixing are inaccurate or cumbersome. If a short-lived radioisotope is introduced and the operation continually monitored by an ion chamber, it is easy to determine when a homogeneous mix is obtained. (See Fig. 6.) Radioisotopes should also prove of value in distillation, drying, evaporation, and other process studies. There are in addition many miscellaneous uses of these radioactive materials; among these are:

1. Organic compounds can be synthesized, incorporating radioactive C^{14} or S^{35} , and the resulting product used as a tracer.
2. Large sources of cobalt (Co^{60}) have been used to radiograph castings, replacing to some extent the more expensive X-ray machines and radium.
3. Radioisotopes have found use in the study of friction and motor wear. Piston rings, after being irradiated in the Oak Ridge National Laboratory reactor, are placed in a normal gasoline engine. The particles of active metal found in the crank-case oil give

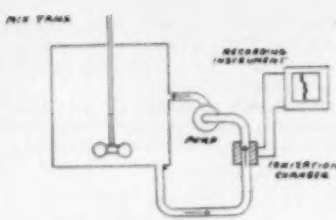


Fig. 6. Monitoring Mix Tank.

Liquid from mix tank is passed through the sample line. Sample line passes through ionization chamber. Mix is homogeneous when radioisotope is uniformly dispersed in carrier.

- data on the rate of wear, etc. With the engine torn down after such a run, X-ray film is placed around the cylinder walls and the points where metal from the ring has migrated to the cylinder detected.
4. Use of a source of radioactivity to dissipate electrostatic charges on heavy machinery has been employed. The radioactive source is placed near the moving parts of the machinery to ionize the surrounding air and thus prevent the build-up of an electrostatic charge. In most cases, an alpha source has been used because of its relatively higher ionizing intensity.
5. Some success has been achieved by using radioactive sources as a thickness gage to measure sheets of plastic or rubber approximately one mil thick. One of the leading rubber companies is using C^{14} as a source of the beta radiation. Other groups are using Sr^{90} or Ru^{106} deposited on metal or ceramic surfaces. The radiation source is placed on one side of the plastic sheet and a sensitive detection instrument located directly opposite on the other side of the film. Some gages reportedly will read with an accuracy of 0.0001 in.
6. Recently neutron sources have been made available for distribution. The neutrons result from the action of gamma rays on beryllium. The gamma rays are emitted from an antimony sphere which has been activated in the pile by neutrons. Approximately 10^6 neutrons/sec. are obtained from the source as it is shipped. The neutron flux drops off with the decay of the Sb^{121} ($T_{1/2} = 60$ d.), but the entire unit can be re-activated in the pile at a cost of \$43/month exposure.

One of these sources is at present being used by the American Museum of Atomic Energy in Oak Ridge to activate dimes for the visitors. One merely drops the dime into a slot at the top of the heavy lead shield. The dime slides to the center of the shield where it is located next to the antimony-beryllium neutron source. In a few seconds, a time release allows the dime to complete its trip through the lead shield to a position over a G-M tube. The blinking lights on the electronic scaler and the click of a counter show that the dime has been activated by the neutrons. A courteous attendant seals the dime into a small aluminum-plastic capsule and the visitor leaves with a harmless souvenir of

his visit to the American Museum of Atomic Energy.

There has been some speculation that these same Sb-Be neutron sources may be of value in some types of process control. In the preparation of a product in which some element appears as a contaminant, neutrons could be used to activate this contaminant which would then be detected by radiation instruments. The contaminant, to be detected satisfactorily, should have a large cross section and a short half-life, as compared to the other elements present.

The distribution of radioisotopes by the Atomic Energy Commission is a growing business. The number of shipments now averages about 520/month or an increase of 150% within the past year. The quantity of radioactive materials distributed, however, has increased by approximately 300% during this same period. The Oak Ridge National Laboratory is continually attempting to improve the quality of the radioisotopes. Many that were originally available only as low specific activity materials (large portion of inactive atoms compared to the small number of active atoms) are now being prepared carrier-free or with an extremely high specific activity. The ratio of active atoms to inactive is important for the successful solution of many problems, particularly some of the biological and medical studies that are being carried on.

Persons interested in obtaining radioisotopes or in discussing a possible application of radioisotopes to their work should write to the Atomic Energy Commission, Isotopes Division, Oak Ridge, Tenn.

Notation

| | |
|-----------|--|
| b | = "barns" = 10^{-24} sq.cm. |
| C | = curies |
| γ | = gamma photon |
| d | = days |
| $dis.$ | = disintegrations |
| e | = base of natural logarithms |
| λ | = decay constant = $\frac{\ln 2}{T_{1/2}}$ |
| | = $\frac{0.693}{T_{1/2}}$ |

| | |
|-----------|--|
| mc | = millicurie |
| mg | = milligram |
| N | = No. of atoms |
| n | = neutron or neutrons/cu.cm. |
| nv | = neutrons passing through 1 sq. cm. within 1 sec. |
| σ | = neutron cross section of a nucleus (sq.cm.) |
| $T_{1/2}$ | = half-life of a radioactive isotope |
| t | = time |
| v | = velocity of neutrons (cm./sec.) |

(Presented at Forty-second Annual Meeting, Pittsburgh, Pa.)

DISCOVER COLUMBUS

Article by R. DAHL

ONE of the gatemen you may see as you walk into Union Station, Columbus, Ohio, providing you're coming to the annual meeting by train—pretty well symbolizes the spirit of this Midwestern metropolis.

This particular gateman—he's tall, thin, and weather-beaten—takes a more than professional interest in getting people on the right train at the right time. People don't have to speak to him first to get help. He's the kind of person who'll walk up to a slightly lost-looking stranger and, in five minutes, ask him where he's going, point out that he's got time for a cup of coffee yet, remind him that he's left his raincoat on the bench, and then bid him goodbye with a sage bit of advice. You'll find a lot of people like the gateman among the more than 500,000 citizens of the Columbus metropolitan area.

Columbus is a modern city—the twenty-eighth largest in the United States—with all the physical advantages of any big city. Yet, its people are friendly. The night after a big football win by Ohio State, the whole town be-

comes one big riotous victory camp. And on Halloween, the downtown area of High Street, one of the two main arteries of Columbus, is roped off for the costumed madness of the thousands of the city's children and young people.

This kind of neighborliness, enthusiasm, and friendliness, coupled with such things as its nationally famous hotels, big-time football and basketball (OSU plays Kansas State Dec. 4 and Butler Dec. 6), Broadway hits at the Hartman Theatre, and other metropolitan offerings has made Columbus one of the top three convention cities in America. Last year, Columbus played host to more than 250,000 people gathering for some 645 conventions.

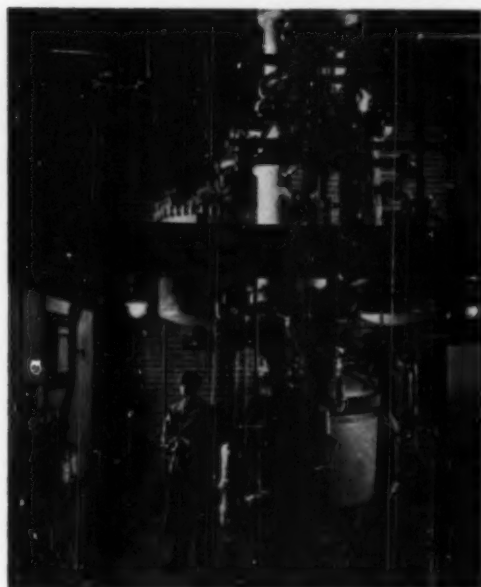
Strategic location is another of the reasons for the town's popularity with conventioners. Columbus' location in almost the exact center of Ohio places it at the hub of the country's most densely populated area. Within a radius of 500 miles of Columbus live almost two thirds of the population of the United States. The country's center of manufacturing, as determined by the

number of wage earners, has been estimated to be within eight miles of Columbus, and the city is only some 200 miles east of the national center of population. This means that the odds are high that most of the A.I.Ch.E. members will travel less than 500 miles to get to the December meeting.

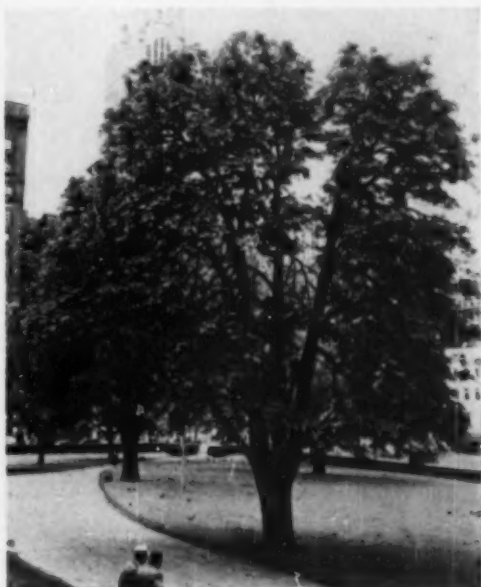
The fact that Columbus is "everybody's" home town is one of the reasons it's becoming known as "Test Town U.S.A."

In recent years, the reaction of Columbus citizenry to a new detergent, an instant coffee, a 60-sec. dessert, and aluminum wrapping to keep foods fresh in the ice box was carefully noted by New York and Chicago advertising executives before they introduced the products to the nation as a whole.

Columbus is rated one of the top six test towns in the nation. Some of the statistical factors considered in the rating: Columbus housewives have a literacy rate 2.5 per cent over the nation's average. They don't fall for duds—are quick to spot a bargain. The average man you see on the street in Columbus



A pilot plant in the chemical engineering laboratories of Battelle Memorial Institute. This two-story apparatus is used in organic synthesis.



The tree for which the state is named—a Buckeye tree in the grounds of the state capitol west of the north entrance.

SOME OF THE LOCAL COMMITTEE CHAIRMEN



Left to right: H. P. Munger, Battelle Memorial Institute, hotels and hospitality; E. A. Beidler, Battelle Memorial Institute, finance, and E. E. Slowter, Battelle Memorial Institute, entertainment.

is making about the same amount of money as the other wage earners of the United States. Last year, Columbus ranked a little over 100th in a list of 200 cities on a per-capita-income basis—\$1475 per year per citizen. Thus, San Francisco, with \$2326-per-capita income, is too high to represent the cities of America as a test area.

In addition, there's a good healthy mixture of white collars and overalls in Columbus. About 10 per cent of the Ohio capital's residents are listed on state, county, or federal payrolls. Columbus has almost 800 manufacturing organizations with around 30 of them listing payroll of more than 500 employees. In addition, more than 9000 railroad men make their homes in Columbus. No one industrial plant dominates the area.

The Pure Oil Co.'s Heath refinery is a good example of the several industries in or near Columbus of particular interest to chemical engineers.

A.I.Ch.E. members visiting the 184 acres of this refinery near Newark, Ohio, will be able to see one of the best-appointed and equipped oil refineries in the Midwest. Its modern facilities produce a wide range of quality petroleum products.

On another plant trip, A.I.Ch.E. members will be able to see the first municipal water-softening plant for a large U. S. city. Built in 1908, its original capacity of 25,000,000 gal./day has since been doubled. The lime-soda process is used in softening the approximately 50,000,000 gal. of water treated/day from a hardness of 232 p.p.m. to 72 p.p.m.

A visit is scheduled to the Mead Corp.'s Chillicothe division, one of the largest plants of its kind in the world. Within the plant, 2200 people work around the clock producing magazine papers, bond papers, and other specialties. More than 100 tons/day of printed wastes are reclaimed with more than

22,000,000 gal. of well water consumed daily.

Other trips will enable visitors to observe the chemical engineering operations used by Columbus manufacturing plants in the production of fertilizers.

Slated for a visit is the Columbus plant of the Smith Agricultural Chemical Co., today a U. S. leader in the fertilizer industry. The plant manufactures sulfuric acid by the chamber process, also superphosphate, farm fertilizers, specialty fertilizers, mineral feeds, and other materials.

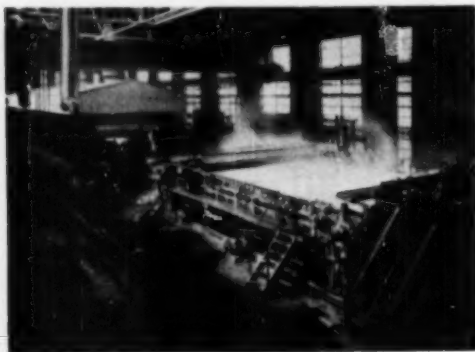
The Farmers Fertilizer Co., also to be visited, has a new chamber process sulfuric acid plant with an annual capacity of approximately 45,000 tons of 60° Baumé acid. These are but a few examples of the industries to be visited during the Columbus meeting.

Ohio State University, Columbus' greatest magnet, drawing people from every section of the country and from abroad, is the fifth largest university in the United States. It has an annual enrollment in normal times of 18,000 men and women students and a faculty of 1,300 members. With more than 1500 students, the OSU Agriculture College is the largest in the world.

Some six universities and colleges, other than the state university, are located in or near Columbus. In such an educational atmosphere, it is only natural to find that research effort has been expanding, not only within the universities but also in the surrounding industries.

One of the important research laboratories of industries to be visited is that of the Owens-Corning Fiberglas Corp., Newark, Ohio. Only 19 years ago, glass fibers were little more than a curiosity, with no significant commercial application. The reason for this situation was high production costs, as well as material limitations in the fibers themselves.


(Continued on page 19)




Formation of paper sheet on a Fourdrinier machine, Mead Corp., Chillicothe, Ohio.



Port Columbus, one of the Midwest's largest airfields, Columbus, Ohio.

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big  expense in gasholder operation

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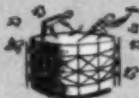


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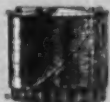


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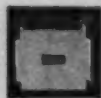
heat



, by ice



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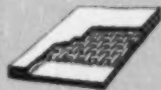


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PROFESSIONAL DEVELOPMENT—THEME OF E.C.P.D. MEETING

LAST month in Cleveland, the Engineers' Council for Professional Development, of which the A.I.Ch.E. is a member, held its eighteenth annual meeting and round-robin discussion of all the phases of professional engineering. Two themes ran through the session: the first, an impending shortage of engineers owing to a greatly decreased enrollment in our colleges and a reactivated Selective Service program. Second, how to instill in the new graduate a sense of professional responsibility. It was this latter idea that saw the Committee on Professional Training present to the delegates a 200-page manual outlining a six-point program for developing a professional outlook during the first five years after graduation in a young engineer's life.

The chairman of the committee, A. C. Monteith, vice-president in charge of engineering and research of the Westinghouse Electric Corp., introduced the report, stressing the fact that our present pace in science and technology is beginning to make itself felt in industry. "Well-trained men are becoming more important than ever in our industrial life," he said, "and the complexities of industry and the increasing tempo of technical development have necessitated a new level of training."

To get better engineers it was the proposal of the group that programs be integrated at a local level to help the young engineer get over the post-college slump to begin in his life a program of professional development which would guide him in the future.

The training program is divided into five parts. The first covers the orientation and training of engineering personnel by employers. The committee studied the training program of 54 companies and reviewed the published information on programs of about 80 others. It found that the cost of industrial training was minor, and that results, after the first year, usually held enough promise to convince management that a continuing program was wise. The committee characterized industrial training as "the first and best opportunity to start the young engineering graduate on a professional development program." The manual contains schedules to help employers to set up effective industrial training programs for engineering graduates.

The second part outlines plans for cooperation in industrial and engineering schools for the continued education of the graduate engineer. "Industrial management must do more than provide

orientation and specialized treatment," the committee report states, "it must be concerned with the education of the whole man, and employers in industry and government, in cooperation with the colleges, have the responsibility of providing continued education for the engineering graduate." A way of meeting this responsibility was suggested by a subcommittee which surveyed study programs and found that out of 50 industrial centers, some 23 offered evening study opportunities through local colleges. For those communities that have no engineering school, they recommended that local engineering societies encourage industry and the state universities to establish extension centers; that the engineering societies sponsor refresher courses, round-table discussions, and lecture series in specialized fields of interest to the community.

The third section of the report dealt with the relationship of the neophyte engineer with his community. The committee said the engineer has a certain responsibility to his community since the public has "made a great investment in him through the taxes and endowment funds provided for higher education."

For guidance in integrating the engineer with the community, the committee surveyed present practices and found no cohesive program. The responsibility, the committee felt, rests first with local sections of engineering societies; second with various community organizations; third with the employer; and fourth with the young engineer himself. Business and service groups were also urged to develop organized programs to welcome newcomers. "Integrating the young engineer into his community will remain a problem only so long as it's a neglected problem. Only a little thought given to its solution in each community will lead to an effective program," the committee concluded.

The fourth part of the volume dealt with engineering registration laws. The viewpoint of the E.C.P.D. committee was that legal recognition does not constitute professional maturity, but rather represents a minimum requirement for engineers. Full professional development depends on continued study and practice of engineering with recognition of responsibility to associates, employer and the public. The committee places on the shoulders of the engineering societies the responsibilities for providing inspiration to carry individuals far beyond the minimum standards

of education and experience which are set by the state.

The final part of the study concerns methods and sample tests to help the young graduate make a self-appraisal of his qualifications so that he can redirect himself to take advantage of the opportunities offered.

A. R. Hellwarth of the Detroit Edison Co. in speaking to the group at the Friday luncheon, asked for better counseling in schools, and to implement such a program he suggested that industry arrange "conferences for teachers from a day to several weeks in length, during which teachers would have all the expenses paid by the sponsoring industry or industries."

The lack of future engineers was first discussed by S. C. Hollister, dean of the college of engineering at Cornell University, who discussed figures that he had taken on behalf of the American Society for Engineering Education, and they are reported in this month's Opinion and Comment.

The same note of impending shortage was struck also by Raymond Walters, president of the University of Cincinnati, at the annual banquet. He said that military preparedness would have "an immediate sharp and devastating effect on higher education. The President's call for 3,000,000 men by the fall of 1951," he said, "may prove to be as serious to higher education as total mobilization in World War II, since the draft is to be of the younger men—19 to 26 years of age. The population in these younger age groups has diminished because of the low birth rate in 1930 and by the exception of veterans and men with dependents." This, he claimed, will result in an increased demand for the college-age youths and he quoted U. S. Commissioner of Education McGrath as saying, "The kind of personnel needed for military duty, young, intelligent, able-bodied men, must be drawn from the age groups normally found in institutions of higher education."

President Walters reported on his survey in which he found that 75% of more than 500 approved colleges and schools have already shown sizable decreases in full-time students as compared with a year ago. Engineering colleges showed heavier losses than other types of colleges both in full-time students and in freshmen, he claimed.

He also stated that a part of the decrease in engineering enrollment is due to a Department of Labor report which indicated that the field of engineering is overcrowded. To combat this idea, he cited the fact that this year's supply of graduates—50,000—a record high, has already been placed in industry.

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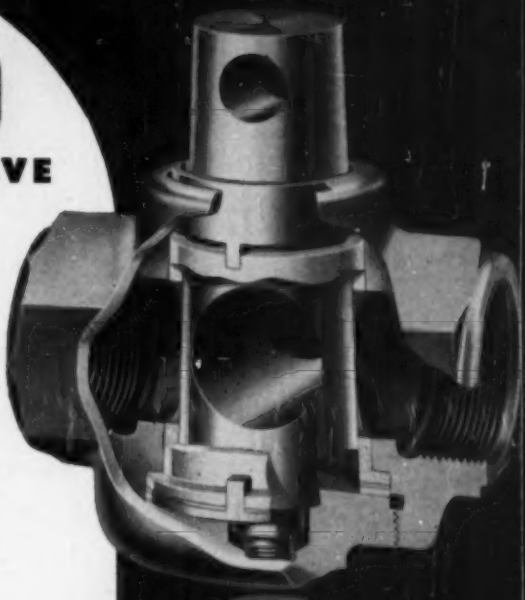
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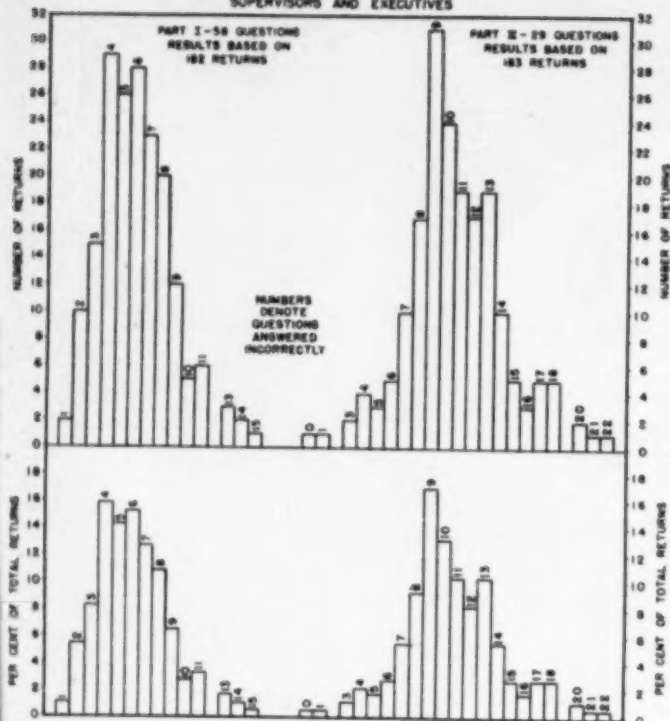
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MINNEAPOLIS RESULTS
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SELF-ANALYSIS QUIZ
FOR
SUPERVISORS AND EXECUTIVES



In the story of the Minneapolis Meeting (see p. 16, October issue), we gave the results of the quiz for supervisors and executives. There we mentioned the fact that chemical engineers had failed to prove that they were either good supervisors or managers. Walter E. Lobo, of The M. W. Kellogg Co., and chairman of the Institute's Program Committee, decided to plot the data to see exactly where the great percentage of mistakes occurred.

The above chart is the result of his work and his Letter to the Editor enclosing it, said in part, as follows:

As you know, according to the directions (of the test) more than three questions missed on Part I indicates 'knowledge of good supervisory practices is on the weak side and needs careful scrutiny.' More than four missed in Part II 'is indicative of weakness in knowledge of managerial principles and in analytical ability.'

Having taken the quiz, I'm not sure that I agree with all the answers—probably many will have the same feeling.

However, be that as it may, the results indicate that about 14% had no more than three wrong in Part I, and 5% no more than four wrong in Part II. If we double the allowable error, for differences of opinion, etc., we find that 60% had no more than six wrong in Part I and 23.5% had no more than eight wrong in Part II. Probably this leniency is excessive.

It would seem reasonable that only about 10% of all chemical engineers might really qualify for managerial duties, and this figure is not too far away from the results obtained.

The test does indicate that a very large percentage is lacking in managerial knowledge—perhaps our programs should try to supply some of this knowledge, since it is not too easy to obtain.

CHEM. ENG. EDUCATION
DISCUSSION IN TORONTO

A program in chemical engineering with a symposium discussion of Chemical Engineering Education, is planned

by the chemical engineering division of The Chemical Institute of Canada. The meeting will take place at the Wallberg Building of the University of Toronto, Feb. 19 and 20, 1951.

This is the first division meeting

sponsored by the chemical engineers of the Institute and the general technical program will feature many papers from engineers both in Canada and the United States.

Prof. R. R. McLaughlin, University of Toronto, is honorary chairman. Prof. W. C. Macdonald, University of Toronto, is chairman of the program committee.

Requests for programs or information should be addressed to A. Monsaroff, Box 6103, Montreal (Que.), Canada.

"CHEMSTRAND" PLANT
FOR MONSANTO-A.V.C.

Plans for the construction of a multi-million dollar plant to manufacture a new synthetic fiber for the Chemstrand Corp. were announced recently by Monsanto Chemical Co. and American Viscose Corp., joint owners of the Chemstrand Corp. Plans call for the plant to be erected on a 656-acre site west of Decatur, Ala., on the Tennessee River. Construction of the plant will get under way next year.

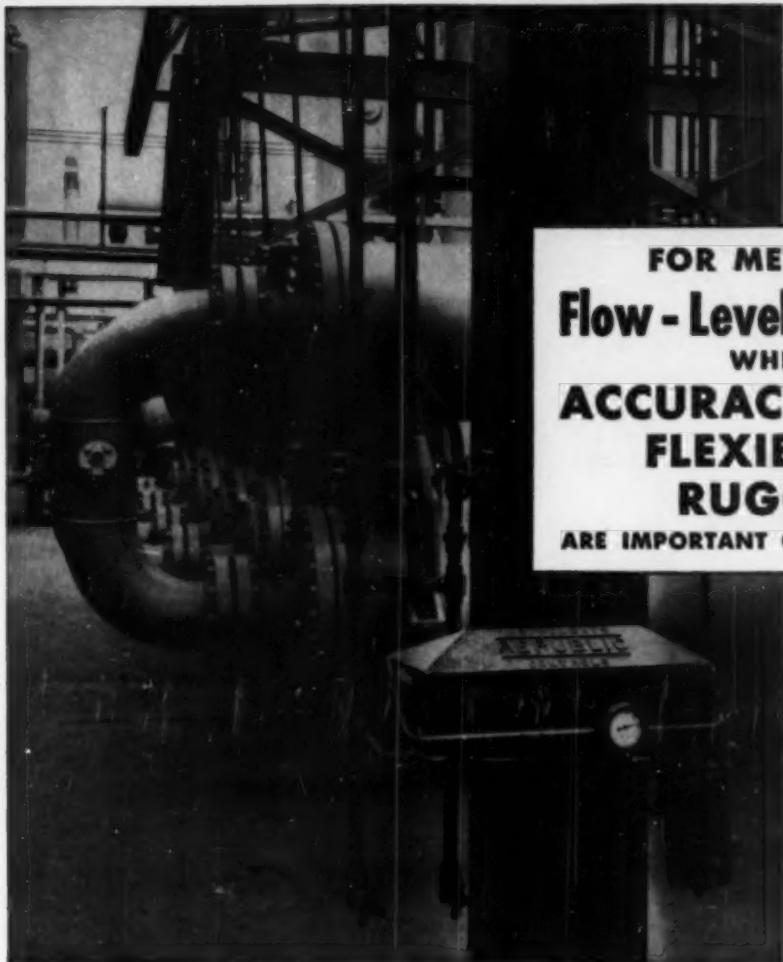
Simultaneously, it was announced that Chemstrand will shortly begin the operation of a synthetic fiber semi-production plant at Marcus Hook, Pa., where a million pounds of synthetic fiber staple will be produced annually. Up until now, the acrylonitrile-type fiber has been produced only in laboratory quantities.

The new synthetic fiber which will be manufactured in staple form in its initial stages, has proved successful in early tests, and, because of its low specific gravity, will tend to offer warmth in light-weight suitings.

Formation of the Chemstrand Corp. was announced May 5, 1949, by Monsanto Chemical Co. and American Viscose Corp. Dr. Carroll A. Hochwalt of St. Louis, a Monsanto vice-president served as temporary president of Chemstrand during the latter's initial research and development stage in the field of synthetic fibers. Osborne Bezanon has been named president. (See People, p. 48.)

The announcement of the Decatur site and Marcus Hook semi-production plant is part of a Monsanto program which announced also the construction of an acrylonitrile plant at Texas City, Tex.

Officers of Chemstrand, in addition to Mr. Bezanon, are Dr. Charles S. Venable of Philadelphia, director of chemical research for American Viscose Corp., who is vice-president of the new corporation, and F. William Koster of Philadelphia, also of American Viscose Corp., who is secretary-treasurer.



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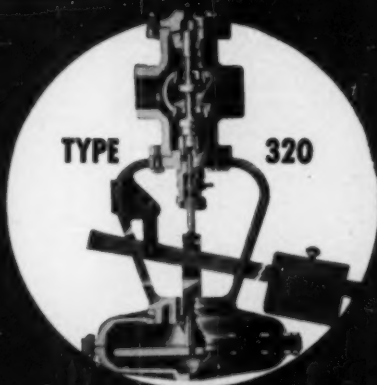
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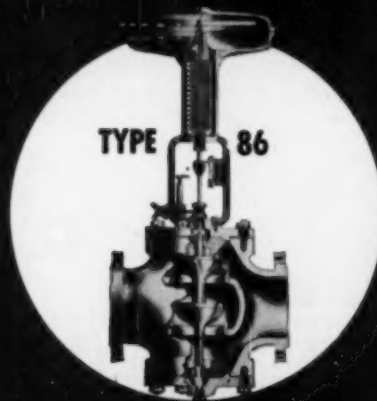
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COLUMBUS STORY

(Continued from page 12)

Most of the research and development work which solved these problems took place in Owens-Corning's Newark research laboratories. Today, Owens-Corning produces glass fibers in seven U. S. and Canadian plants and more than 6000 U. S. employees alone are making hundreds of fiber glass products for everyday commercial use. Within Columbus and Central Ohio, numerous other laboratories for industrial research are playing an important role in America's technological progress.

The Research Foundation on the Ohio State University campus administers contract research sponsored by industry and government. Research projects are conducted in the university laboratories and the Engineering Experiment Station. Faculty supervision provides high-quality training of men for research under this system.

The Ohio State University's chemical engineering department has an active part in administering the work of the Research Foundation, in addition to promoting research under private fellowship grants.

A.I.Ch.E. members scheduling a plant trip to the Ohio State University will see scientific points of interest such as (1) Cryogenics Laboratories—for the production and study of liquid air, hydrogen, and the isotopes of nitrogen; (2) American Petroleum Institute Laboratories where pure hydrocarbons are made; (3) research on materials of construction and corrosion; and (4) chemical engineering laboratories—industrial process development, chemical engineering operations, and thermodynamic investigations.

Near the University campus is located Battelle Institute, one of the world's leading research organizations. Its staff of 1300 technologists and assistants conducts research for the benefit of both private industry and government agencies. Chemical engineers are active in nearly all of the 33 research divisions. Visitors can observe equipment for the study of most of the chemical engineering operations and processes. There are specialized laboratories for assistance to industries in fields of rubber, paints, plastics, fuels, ceramics, corrosion, electroplating, high-pressure techniques, air and stream pollution control, metallurgy, and mineral processing.

Another research center which will be visited is the Edward Orton, Jr., Ceramic Foundation. Applied research in the ceramic field is conducted, in addition to the manufacture of pyrometric cones.

(Continued on page 40)

DOW'S NEW AMMONIA PLANT IN TEXAS



Above is shown the new plant of The Dow Chemical Co. at Freeport, Tex., for the production of refrigeration-grade and technical-grade anhydrous ammonia. The anhydrous ammonia will be used for direct fertilization of soil in considerable quantity on the West Coast and in the Delta region of Arkansas, Louisiana, and Mississippi, and for usual industrial applications.

R. E. DOHERTY DIES

Robert Ernest Doherty, who retired from the Carnegie Institute of Technology presidency in July of this year, died Oct. 19, in Scotia, N. Y. He was 65.

During his 14-year tenure at Carnegie, Dr. Doherty made one of the greatest contributions of this century to undergraduate professional education in his creation and development of what has since become known as the Carnegie Plan. In addition to his educational pioneering, Dr. Doherty more than doubled the Carnegie endowment and carried through major administrative reorganizational programs.

Born in Clay City, Illinois, Jan. 22, 1885, Dr. Doherty was educated at the Academy of the University of Illinois and at the University, where he won a B.S. degree in 1909.

After graduation, he worked for the General Electric Co. as a test engineer (1909-1910); as a design engineer (1910-1918); and as assistant to Dr. Charles P. Steinmetz (1918-1923). He organized the advanced course in engineering offered by GE; and in 1931, Yale University called him as professor and chairman of the department of electrical engineering. He became dean of the Yale School of Engineering in 1933, a post he held until 1936 when he be-

came third president of Carnegie.

He was one-time president of the Society for the Promotion of Engineering Education and chairman of the Engineers' Council for Professional Development.

MEMPHIS PLANT FOR DU PONT CO.

Plans for the construction of a chemical plant at Memphis, Tenn., at an estimated cost of \$7,500,000, were announced recently by the Du Pont Co. The new plant will be built about 10 miles north of the business district on a 225-acre site purchased last May and will be operated by the company's electrochemicals department.

Sodium cyanide will be manufactured at the plant which will employ about 180 persons. The new plant will be of the open-air type of construction conforming to the latest advances in chemical process design. Most of the equipment will operate in the open with controls located in small buildings.

STUDENT ACTIVITIES OF LOCAL SECTIONS GROW

A recent survey of local section activities by J. J. Healy, Jr., chairman of the Institute Committee on Local Sections

has revealed that at least five sections have programs designed to encourage student activities in chemical engineering in their respective areas. Some of these programs have been of long standing; some have only recently been organized. All of them are slanted toward providing the student chemical engineer with recognition by practicing members of the profession.

The subject will come under discussion by the Local Section Committee at the Columbus annual meeting, and it is hoped that the effort will show local sections the value of undertaking co-operative programs with A.I.Ch.E. Student Chapters. The purpose of the round-table discussion is to induce local sections within the Institute to introduce such programs within their sections.

According to the report of J. J. Healy, the sections with positive student programs are Philadelphia-Wilmington, Rochester, Louisville, Ohio Valley, and the Rocky Mountain Sections.

The Fred C. Zeisburg Memorial Award administered by the Philadelphia-Wilmington Section is perhaps the oldest of the local sections-student chapter cooperative programs which has remained continuously in effect. This award, established in 1939, is for excellence in technical report writing by senior students in chemical engineering. Students from eight engineering schools participate in the contest. Their reports are judged by a committee, and cash awards are presented to the winners at the April meeting of the section.

In Rochester, the local section holds annually a meeting at the University of Rochester with the student chapter having entire charge of the gathering, including the arrangements for the speaker. A Student Sponsorship and Professional Guidance Committee has been the element responsible for increasing student attendance at Louisville Section meetings and in making the students feel completely welcome. "University of Cincinnati Night" is the title of an annual feature of the Ohio Valley Section's meeting program. Undergraduate and graduate students present brief technical papers and are the guests of the section at the dinner which precedes the talks. Early this year, the Rocky Mountain Section initiated a "Student-Meeting-in-Miniature" with chemical engineering students from the University of Colorado and the University of Denver competing in oral and written presentations. The meeting was held on the campus of the University of Denver. In concluding his report, Mr. Healy asked that officers of local sections contemplating student programs attend the Columbus meeting of his committee and participate in the planning.

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MARGINAL NOTES

News of Books of Interest to Chemical Engineers

Simplified Treatment of Coal Problems

Coal, Coke, and Coal Chemicals. First Edition. Philip J. Wilson, Jr., and Joseph H. Wells. McGraw-Hill Book Co., Inc., New York, N. Y. (1950) 509 pp., 97 tables, 228 illustrations. \$9.00.

Reviewed by I. D. Clendenin, U. S. Bureau of Mines, Schuylkill Haven, Pa.

THIS well illustrated volume should be a useful addition to the library of many engineers and technologists who deal with coal. The author has purposefully simplified his treatment of various topics for the benefit of students and those interested persons who are less familiar with the details of coal properties and behavior, coal carbonization and the coking industry.

This treatise complements "Chemistry of Coal Utilization," now a much-used reference work which was edited by H. H. Lowry and published in 1945. The authors recognized and have filled a long-standing need for an elementary text covering both modern commercial practice and technological developments in coal carbonization.

Following the introductory chapter in which the nature and scope of coal carbonization are outlined, the second chapter is devoted to a brief discussion of fuels and combustion.

The origin, classification and properties of coals, their characteristics for carbonization and preparation, cleaning and storage of coal for coking are well presented in the next three chapters. The discussion of the coalification process and elementary composition and analysis is appropriate.

The subject of assaying, testing and selecting coals for carbonization is abridged, but with sufficient completeness to make the reader cognizant of the problems in coal testing and selection. In the discussion of "swelling during coking," a subject whereof some diversity of opinion exists, a misunderstanding may arise because the empirical test results which are described for the "constant volume" and "constant pressure" methods are not clearly differentiated. One might add that the maximum free-swelling index of 9 need not mean that no coal will give a greater index. Early in this chapter the authors bring out the irreversibility of the fusion or plastic behavior of coals that are coking or exhibit "fusion." This fact is not too well appreciated.

Three chapters are given to the subjects—Coke Ovens, The Coking Process in By-Product Coke Ovens, and High-Temperature Coke—while the next four chapters are devoted to gas and coal chemicals, their recovery, separation, purification, handling and storage. Gas, Ammonia and Ammonium Salts, Light Oil and Coal Tar are each discussed in separate chapters. There is a nice discrimination in the presentation of details of design, recovery, purification, waste-handling and related principles, without burdening the reader with details in a field which is the authors' forte.

The final two chapters on the technology of carbonization include a discussion of "Coal Carbonization for Gas Manufacture" and a description of Some Low-Temperature Carbonization Processes and the Curran-Knowles Process. Although the proportion of coke, gas and coal chemicals being produced commercially in gas ovens and retorts and low- and medium-temperature processes is small (less than 2% of total coal carbonized in 1946), these methods merit attention.

The concluding chapters treat of the economics and trends in the coal-carbonization industry. The authors have considered the use, relative value and comparative importance of the various products.

The list of visual aids at the end of the book will be appreciated and the too select bibliography in Appendix II should prove valuable.

Few typographical or proofreading errors were noted. On page 231 specific gravity has the units "% by weight." On page 447, a value of 8 per cent is used when 3 per cent appears in the table on the same page.

An Authoritative Listing

Electronic Engineering Master Index 1949. Electronics Research Publishing Co., Inc., New York, N. Y. Editor, John F. Rider. (1949) 296 pp. + xvi. \$17.50.

THIS is a bibliographical listing of research on electronics, atomics, optics, physics and allied fields. This 1949 edition contains 12,500 references from more than 400 world-wide publications, U. S., Canadian, and British declassified documents, and U. S. patents; more than 600 authoritative subject headings; cumulative subject cross index covering 1925-45, 1946, 1947-48 and 1949 editions also included.

Thermodynamics for Chemical Engineers and Chemists

Chemical Thermodynamics. First Edition. Frederick D. Rossini. John Wiley & Sons, Inc., New York, N. Y. (1950) 514 pp. \$6.00.

Reviewed by James Conll, Professor and Head, Chemical Engineering Department, University of Pittsburgh, Pittsburgh, Pa.

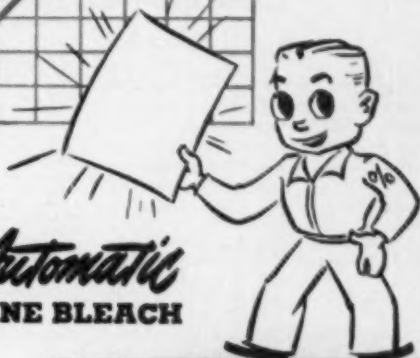
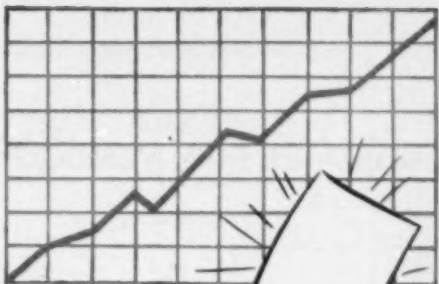
PROFESSOR ROSSINI holds a distinguished position as a contributor to the literature of thermodynamics, notably in formulating thermochemical standards for a large number of pure hydrocarbons. His work at the National Bureau of Standards was not confined to laboratory and conference room but also included the teaching of thermodynamics to graduate students.

Chemical engineers who pride themselves on their definitions should read this book, especially the chapter devoted to a discussion of the calorie and the joule. The author pin-points his definitions; and because the various symbols are defined as they are introduced, the book is easy to read. It is fitting, in view of the dedicatory preface to G. N. Lewis, that nomenclature should follow the Lewis and Randall system, although one sees a more liberal use of superscript and subscript. If one were to characterize the book as a proper sequel to the famous Lewis and Randall text, injustice might be done to its authorship. It is a distinct contribution on its own merit.

It is interesting to compare the divergent treatment of thermodynamics as presented to the chemist and chemical engineer. This book opens new frontiers, especially in laying a more exact foundation for an understanding of entropy and the third law. The groundwork is laid in the statistical calculations of thermodynamic functions which this reviewer considers to be a high point of the book. This treatment prepares the reader to consider a number of examples in apparent violation of the third law. Another category of interest not usually considered in chemical engineering treatments is that of electrolytic solutions. There is presented in this volume a practical treatment of the thermodynamic implications of the Debye-Huckel theory.

(Continued on page 37)

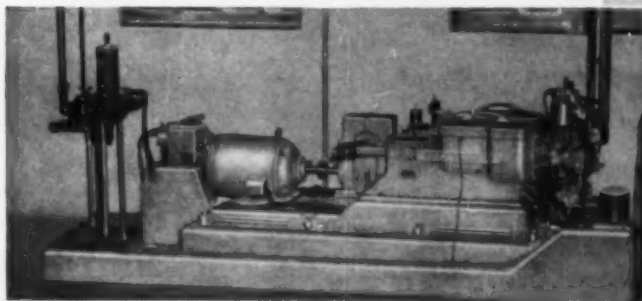
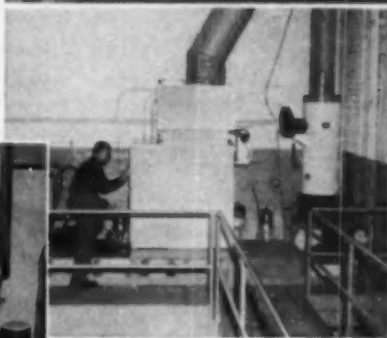
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The following is a list of candidates for the designated grades of membership in A.I.Ch.E. recommended for election by the Committee on Admissions.

These names are listed in accordance with Article III, Section 7, of the Constitution of A.I.Ch.E. which states:

Election to membership shall be by vote of the Council upon recommendation of the Com-

mittee on Admissions. The names of all applicants who have been approved as candidates by the Committee on Admissions, other than those of applicants for Student membership, shall be listed in an official publication of the Institute. If no objection is received in writing by the Secretary within thirty days after the mailing date of the publication, they may be declared elected by vote of Council. If an objection to the election of any candidate is received by the Secretary within the period specified, said objection shall be referred to the Committee on Admissions, which shall investigate the cause for such objection, holding all communications

in confidence, and make recommendations to the Council regarding the candidate.

Objections to the election of any of these candidates from Active Members will receive careful consideration if received before Dec. 15, 1950, at the Office of the Secretary, American Institute of Chemical Engineers, 120 East 41st St., New York 17, N. Y.

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H. A. Holcomb, *Austin, Tex.*
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Ernest W. Thiele, *Chicago, Ill.*
Arthur C. Thomas, *Chicago, Ill.*
Benjamin Thompson, *Kingsport, Tenn.*
F. M. Tiller, *Nashville, Tenn.*
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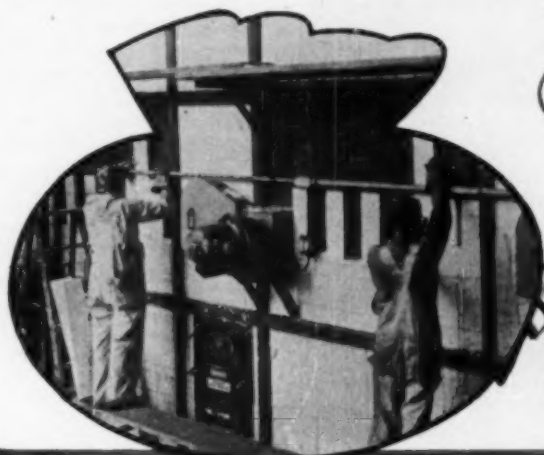
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Melvin J. Beagle, Jr., *Harvey, Ill.*
Carol E. Belton, *Texas City, Tex.*
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Carey W. Brackin, *Whiting, Ind.*
Robert Edward Branch, Jr., *Little Rock, Ark.*
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Ben H. Loper, *Stamford, Conn.*
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Francis Pall, *South Bend, Ind.*
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Charles D. Price, *Austin, Tex.*
Charles Robert Quinn, *Buffalo, N. Y.*
Glenn Ralston, *Relay, Md.*
George A. Rees, *Oak Park, Ill.*
Lester Jay Reynolds, Jr., *Rochester, Pa.*
Harrison B. Rhodes, *Marcus Hook, Pa.*
Frank E. Rush, Jr., *Wilmington, Del.*
Rodney G. Schroeder, *Huntington, W. Va.*
Herbert C. Scribner, *Oldtown, Me.*
Lawrence H. Shenker, *Bayside, N. Y.*
Stuart A. G. Singer, *Wilmington, Del.*
Walter E. Smith, Jr., *Kala, Hawaii*
Oddvar Solstad, *Medford, Mass.*
Norman Stein, *Wilmington, Del.*
Arthur H. Vogt, *Matawan, N. J.*
Clayton R. Wagner, *Panama, N. Y.*
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LOCAL SECTION NEWS

SPEAKERS AT FOURTH OKLAHOMA STATE MEETING



The Oklahoma State Meeting of A.I.Ch.E. was held in Tulsa, Oct. 7, on the University of Tulsa campus. There are two local sections of the Institute in Oklahoma which alternate in sponsoring these one-day meetings. A registration of 175 marked the attendance for the all-day meeting sponsored this year by the Tulsa Section.

This was the Fourth Annual Oklahoma State Meeting of A.I.Ch.E.

The technical program this year was divided into three sessions as follows: Natural Gas Liquids (W. L. Nelson, presiding); Refining (J. E. Swearingen, presiding); and Gas Products (C. W. Perry, presiding).

Above shown from left to right are the speakers: J. W. Jewell, Jr., The M. W. Kellogg Co., "Economics of Petroleum Coking"; A. L. Kohl, The Fluor Corp., Ltd., "The Selective Removal of Hydrogen Sulfide from Gas Streams"; V. E. Middlebrook, consultant, "Natural Gas Processing"; Vladimir Haensel, Universal Oil Products Co., "Platforming"; W. T. Tierney, The Texas Co., "Texaco Combustion Process"; T. P. Latimer, Clark Bros., Inc., "Centrifugal versus Reciprocating Compressors"; M. Webb, Stanolind Oil and Gas Co., "The Production of Sulfur from Hydrogen Sulfide," and B. R. Carney, Warren Petroleum Co., "Natural Gas Liquids."

OHIO VALLEY

A meeting was held Oct. 2 at the Herman Schneider Foundation Building in Cincinnati. J. C. Frommer, consulting engineer associated with the Electric Eye Equipment Co., Danville, Ill., gave a review of the application of electronically operated controls for process work. He discussed and demonstrated the "Purifil" control for determining cloudiness in fluids.

On Nov. 6 Guy N. Harcourt, vice-president of engineering, Buřlovak equipment division, Blaw Knox Co., spoke on "Vacuum Drum Drying."

Reported by R. W. Evans

ROCHESTER

The first meeting of the year was held Oct. 18, 1950, at Rundel Library.

The speaker for the evening was Dr. Warren McCabe, President of A.I.Ch.E. Dr. McCabe talked on the subject—"A.I.Ch.E.—1950 Model," and explained in some detail the general operation of the Institute. He also discussed the publication, *Chemical Engineering Progress*, its quality and number of papers printed.

Reported by O. J. Britton

ST. LOUIS

At the Oct. 17 meeting at the York Hotel, with 100 members and guests in attendance, Albright of Socony-Vacuum Oil Co. presented a paper on "Gas Turbine Fuels." He said this is an important present-day subject since military aircraft requires special-type fuels for jet operation. The first type of fuel developed was a kerosene-base type, however, it was limited in quantities which could be produced and its freezing temperature was too high. The now standard jet fuel, a mixture of light fuel oils and gasoline, has a low freezing temperature and is available in quantities which will surpass by 500 per cent the quantities of high octane fuel produced for the military service by petroleum refineries in the last war. After these remarks he discussed the present standard jet fuel under the following operating factors: handling and pumping, starting, carbon deposition, combustion efficiency, flame stability, and range and payload.

The fall activities began Sept. 13 with a "smoker" held in the reception hall and dining room of the Bevo Building, Anheuser-Busch. Approximately 95 members and guests were in attendance. No formal program was presented; the members were entertained by the Mon-

santo Research Philharmonic Orchestra who played through most of the evening.

Reported by R. S. Yates

TERRE HAUTE CHEMICAL ENGINEERS' CLUB

The chemical engineers of the Terre Haute (Ind.) area were called together the evening of Oct. 10, in a meeting to organize the Terre Haute Chemical Engineers. It is the intent of this club to qualify for a charter as a Local Section of A.I.Ch.E.

The meeting was attended by 47 area engineers.

The following officers were elected for the current year.

Chairman—W. S. Kaghan, Rose Polytechnic Institute

Vice-Chairman—A. C. Miller, Commercial Solvents Corp.

Secretary—E. J. Massaglia, Chas. Pfizer & Co.

Treasurer—Eli Zinn, Commercial Solvents Corp.

Executive Committee—S. G. Bankoff, R. S. Egly, W. E. McCoy

Reported by E. J. Massaglia

EAST TENNESSEE

At the meeting on Sept. 19, held in the foremen's conference room of the Tennessee Eastman Corp., Kingsport, Tenn., about 26 members and guests were present.

The speaker of the evening was O. J. Whittamore, a ceramics research engineer connected with the Norton Co. at Worcester, Mass. His subject was "Pure Oxide Refractories." Mr. Whittamore illustrated his discussion with slides.

Reported by E. L. Gustafson and R. H. Morrison, Jr.

LOUISVILLE

The first fall meeting of this section was held on Oct. 10 in the Speed Auditorium. The speaker, L. S. Stinson, discussed "Steam Jet Air Ejectors for the Chemical Industry." Mr. Stinson is the field engineer at Cincinnati for the Elliott Co.

Reported by W. B. Altshuler

COLUMBIA VALLEY

The first fall meeting of this section held Sept. 28 in Richland, Wash., was addressed by J. H. Hull, manager of process development, Crown-Zellerbach Corp. In his talk entitled, "Problems of Waste Disposal from Sulfite and Sulfate Pulp and Paper Mills," he reviewed various waste treatment methods, and outlined the newly developed ammonia-base pulping procedures.

Reported by George Sege

(Continued on page 28)

SECRETARY'S REPORT

S. L. TYLER

THE Executive Committee of the Institute met at the Institute office in New York, Oct. 6, 1950. The Minutes of the September meeting were read, Treasurer's report and bills received and approved. All candidates for membership whose names appeared in C.E.P. for September, 1950, were elected to membership.

The following were appointed as counselors of student chapters as indicated: A. X. Schmidt at the College of the City of New York to succeed M. Kolodney and C. J. Major at the State University of Iowa to succeed J. O. Osburn. W. Edgar Gift, O. L. Updike and H. Zabel were added to the Professional Guidance Committee at the request of P. D. V. Manning, chairman. A. Syversen was added to the Public Relations Committee at the request of J. H. Perry, chairman. Other routine matters dealt with membership or changes in membership status.

The Council met at The Chemists' Club in New York City Oct. 6. Secretary presented request from the National Security Resources Board through Engineers Joint Council to appoint three members of an advisory committee to the Manpower Division of NSRB to act along with similar advisory groups from the American Chemical Society, American Institute of Physics, and the National Research Council. The appointees were: F. J. Curtis, C. G. Kirkbride and W. I. Burt.

The budget for 1950 was reviewed in the light of experience for the greater portion of the year and it was found to be in order.

Upon recommendation of J. J. Healy, Jr., chairman, Local Sections Committee, the applications for local sections were approved as follows: the National Capital Section at Washington, D. C., the Central Virginia Section and the Sabine Area Section.

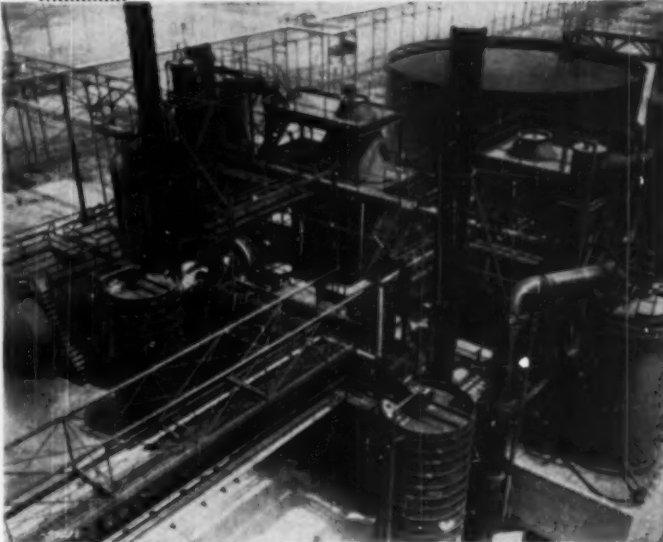
A preliminary draft of new bylaws was submitted by the Constitution and Bylaws Committee and discussed at some length. No definite actions were taken regarding these bylaws but study will be continued.

The report of the Chemical Engineering Education and Accrediting Committee was submitted and approved.

Committee chairmen were appointed for the year 1951 and are to be advised of their appointments and asked for recommendations as to committee personnel in order that committee appointments may be completed by the first of the year.

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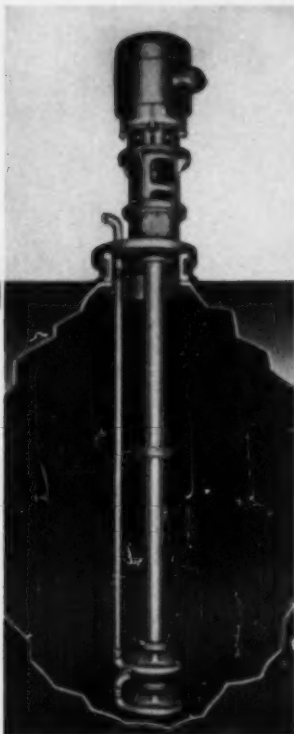
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LOCAL SECTION CALENDAR

CHARLESTON SECTION: Nov. 21, Charleston Recreation Center. R. L. DODGE, DU PONT CO.—"Some Important Factors in Plant Location."

CHICAGO SECTION: Dec. 20, Western Society of Engineers suite. F. W. KLAY, J. E. SEAGRAM & SONS—"Quality Control."

MARYLAND SECTION: Dec. 12, Johns Hopkins University, Levering Hall Cafeteria. G. E. MULLIN, DOWELL, INC. — "Combating Corrosion with Metal From the Sea."

NEW JERSEY SECTION: Dec. 12, Public Service Auditorium, Newark. A. L. BAKER, KELLEX CORP.—"Progress in the Atomic Energy Field."

NEW YORK SECTION: Dec. 13, Brass Rail, Fifth Ave. and 43rd St. Joint Student Chapter meeting. Panel discussion "On the Job Training."

OHIO VALLEY: Nov. 29, Inspection Trip of the Gulf Refining Co., Cleves, Ohio, at 7:30 P.M. Dec. 15, Herman Schneider Foundation Building, Cincinnati, Ohio. R. C. GUNNESS, STANDARD OIL CO. OF INDIANA.

ROCKY MOUNTAIN SECTION: Dec. 8, V. L. MATTSON, COLORADO SCHOOL OF MINES—"Flotation."

MARYLAND

The second meeting this fall was held Nov. 15 at Johns Hopkins University. The lecture at Maryland Hall was given by G. O. Thrift of the Koch Engineering Co. He discussed "The Kaskade Tray and Its Relation to Bubble Trays."

The first meeting was held Oct. 10 at Johns Hopkins University. The speaker, G. N. Harcourt, Blaw-Knox Construction Co., discussed history, development and operation of vacuum drum dryers.

Reported by Alan Beerbower

NEW ORLEANS

At the Oct. 19 meeting held at the St. Charles Hotel, D. V. Probasco of the Wellington Sears Co., New York, discussed the development of Lantuck, a resin-bonded nonwoven fabric, covering its history from its inception through the laboratory, pilot plant and production stages.

Reported by R. M. Persell



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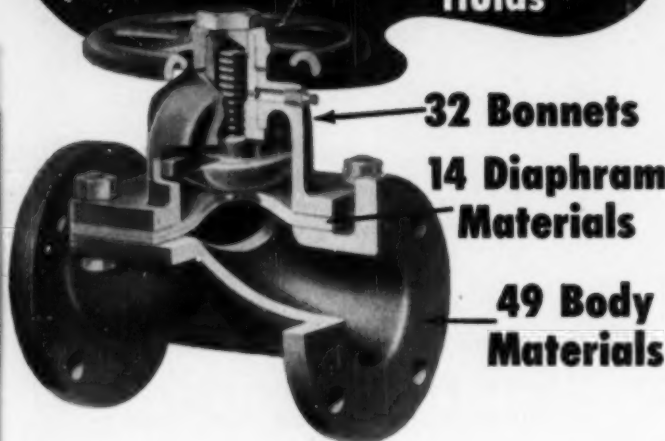
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NORTHERN CALIFORNIA

This section met at the Engineers' Club in San Francisco, Oct. 2. There was an attendance of 85. Speaker at the dinner was John M. Dodds, who is associated with the apparatus department of General Electric Co. in San Francisco.

Mr. Dodds' subject was "Jet Propulsion." He discussed the various phases of the subject and briefly described engine construction details, operating pressures and temperatures and fuels.

Reported by J. V. Hightower

DETROIT JUNIOR GROUP

At the annual business meeting Oct. 4, 1950, in the Junior Room of the Rackham Memorial, two sound films dealing with atomic energy were shown. They were, "Atomic Power," a March of Time release, and "Operation Crossroads," the official color record of the atomic explosions at Bikini. Following the showing, Paul Lewis, Wyandotte Chemicals Corp., reported on the main points of his conversation with Brig.-Gen. Clyde E. Dougherty, director of Detroit's Office of Civilian Defense, with respect to the possible aid chemical engineers can contribute in the event of an atomic attack.

Reported by W. W. Jones

PITTSBURGH

Warren L. McCabe, former head of the department of chemical engineering, Carnegie Institute of Technology, and now vice-president of Flintkote Co., was the speaker of the evening at the opening dinner meeting of the 1950-51 season, held Oct. 4 at the College Club. The incoming chairman of the local section, Carl C. Monrad, introduced the speaker, whose address was entitled, "The Institute, 1950 Model." Dr. McCabe is President of A.I.Ch.E.

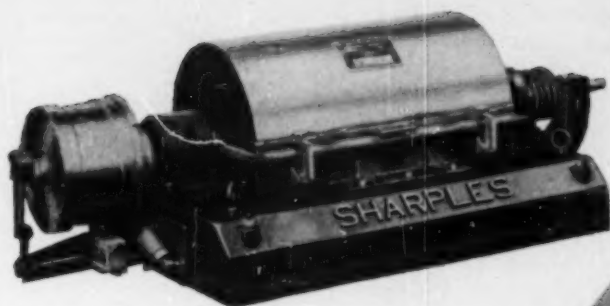
Dr. McCabe gave an outline of the organization and functions of A.I.Ch.E., with regard to national and local section operations. His remarks covered four general topics: (1) method of operation of A.I.Ch.E.—constitution, charter, bylaws, standing committees; (2) membership—present status and future trends; (3) role of local sections, and suggested activities; (4) publications—present status, and future possibilities.

Reported by Hugh L. Kellner

CHICAGO

The first regular meeting of the fall season was held at the Western Society of Engineers suite Oct. 18 with about 120 present for dinner. J. M. Gillet of the Victor Chemical Co. gave a talk on "Market Research in Theory and Practice."

Reported by Thorpe Dresser

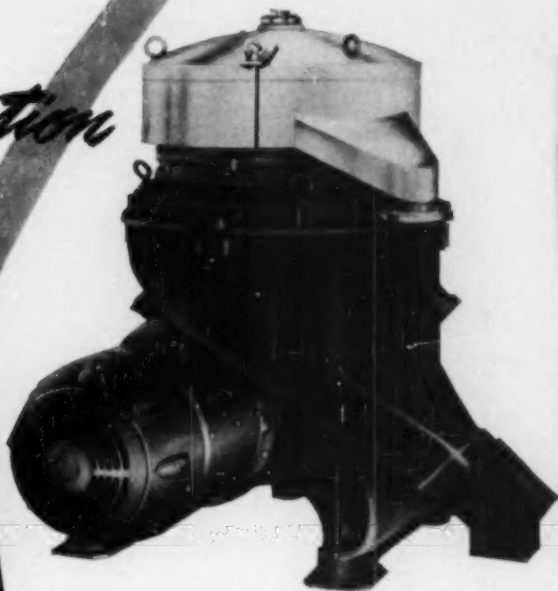


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Chairman of the A.I.Ch.E. Program Committee

Walter E. Lobo

The M. W. Kellogg Co.,

225 Broadway, New York 7, N. Y.

MEETINGS

Annual—Columbus, Ohio, Neil House, Dec. 3-6, 1950.

Technical Program Chairman: John Clegg, Battelle Memorial Institute, Columbus, Ohio

Regional—White Sulphur Springs, W. Va., The Greenbrier, March 11-14, 1951.

Technical Program Chairman: Walter E. Lobo, The M. W. Kellogg Co., 225 Broadway, New York, N. Y.

Regional—Kansas City, Mo., Hotel Muehlebach, May 13-16, 1951.

Technical Program Chairman: Walter W. Deschuer, J. F. Pritchard Co., Kansas City, Mo.

Regional—Rochester, N. Y., Sheraton Hotel, Sept. 16-19, 1951.

Annual—Atlantic City, N. J., Chalfonte-Haddon Hall Hotel, Dec. 2-5, 1951.

Technical Program Chairman: Frank J. Smith, Pan American Corp., New York, N. Y.

Regional—French Lick, Ind., French Lick Springs Hotel, May 11-14, 1952.

Technical Program Chairman: W. W. Kraft, The Lummus Co., 385 Madison Avenue, New York, N. Y.

Authors wishing to present papers at a scheduled meeting of the A.I.Ch.E. should first query the Chairman of the A.I.Ch.E. Program Committee, Walter E. Lobo, with a carbon copy of the letter to the Technical Program Chairman of the meeting at which the author wishes to present the paper. Another carbon should go to the Editor, F. J. Van Antwerpen, 120 East 41st Street, New York 17, N. Y. If the paper is suitable for a symposium, a carbon of the letter should go to the Chairman of the Symposia, instead of the Chairman of the Technical Program, since symposia are not scheduled for any meeting until they are complete and approved by the national Program Committee. Before authors begin their manuscripts they should obtain from the meeting Chairman a copy of the Guide to Authors, and a copy of the Guide to Speakers. The first book covers the preparation of manuscripts, and the second covers the proper presentation of papers at A.I.Ch.E. meetings. Presentations of papers are judged at every meeting and an award is made to the speaker who delivers his paper in the best manner. Winners are announced in *Chemical Engineering Progress*, and a scroll is presented to the winning author at a meeting of his local section. Since five copies of the manuscript must be prepared, one should be sent to the Chairman of the symposium and one to the Technical Program Chairman of the meeting, or two to the Technical Program Chairman if no symposium is involved and the other three copies should be sent to the Editor's office. Manuscripts not received 70 days before a meeting cannot be considered.

SYMPOSIA

Air and Water Pollution Control
Chairman: Richard D. Hoak, Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh, Pa.

Meeting—Columbus, Ohio

Chemical Engineering in Glass Industry

Chairman: F. C. Flint, Hazel-Atlas Glass Co., Washington, Pa.

Meeting—Columbus, Ohio

Processing of Viscous Materials
Chairman: W. W. Kraft, The Lummus Co., 420 Lexington Ave., New York, N. Y.

Meeting—Columbus, Ohio

Phase Equilibria

Chairman: W. C. Edmister, Carnegie Inst. of Tech., Pittsburgh, Pa.

Meeting—Columbus, Ohio

Relationship Between Pilot-Scale and Commercial Chemical Engineering Equipment

Chairman: Walter E. Lobo, The M. W. Kellogg Co., 225 Broadway, New York, N. Y.

Meeting—White Sulphur Springs, W. Va.

Vacuum Engineering

Chairman: W. W. Kraft, The Lummus Co., 385 Madison Avenue, New York, N. Y.

Meeting—French Lick, Ind.

for the coming year. He also announced that 12 of the local club's membership had filed applications for membership in A.I.Ch.E. as a result of the membership drive conducted during the summer months.

Reported by E. D. Wurster

NEW YORK

At the Management Symposium (see October issue, p. 30) of this section held Oct. 24 at the Statler Hotel, John E. McKeen, president of Charles Pfizer & Co., Inc., formally announced the discovery of two new antibiotics—Netropsin and Thiolutin. This makes



E. L. Demarest
Blaw-Knox Co.,
chairman,
Management
Symposium

four new antibiotics discovered by Pfizer in the past 12 months, the other two being Terramycin and Viomycin. Reference to these discoveries was correlated with Mr. McKeen's talk on "The Functions of Management."

Dr. Robert E. Wilson, chairman of the board, Standard Oil Co. of Indiana, was the dinner speaker. In his talk on "Strategy for True Liberals," he told in no uncertain terms, how we need to set up a strategy to combat the growing menace of government controls. He then outlined twelve points of strategy to further the ultimate aim.

A luncheon meeting at the Brass Rail Restaurant Nov. 15 featured a paper by David E. Pierce of General Aniline and Film Corp. entitled, "Maintenance."

Reported by L. C. Johnston

CHARLESTON, W. VA.

A general meeting was held at the Charleston Recreation Center, Oct. 17, 1950, with 175 members and guests present.

S. L. Tyler, A.I.Ch.E. Executive Secretary, spoke briefly on the current technical manpower situation.

The speaker of the evening was Murray Raney, president of the Raney Catalyst Co. His subject was "Hydrogenation Catalysts." Mr. Raney introduced his subject by pointing out how inexact is the science of catalysis. He pointed out that no tables of data, no calculations, are generally applicable. He likened a catalyst to a tool which the chemist can use and construct, but cannot design.

Reported by M. C. Guthrie, Jr.

EL DORADO CHEMICAL ENGINEER'S CLUB

Fifty-five members and guests were addressed by J. E. Vassie, technical director for the International Paper Co. at its Cullendale (Ark.) plant, at the club's first dinner meeting of the fall

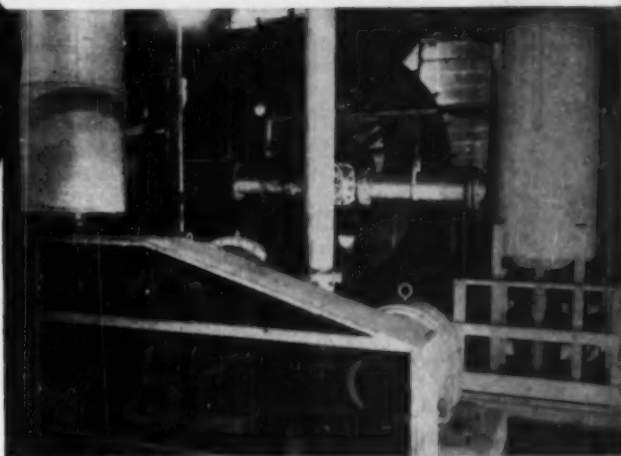
season held in the Garrett Hotel Sept. 29. The subject of Mr. Vassie's talk was "Manufacture of Kraft Pulp and Paper."

Prior to Mr. Vassie's address, Dr. Charles Davenport, president of the club, told the members of plans for nomination and election of new officers

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EQUIPMENT

1 • MIDGET BELT FEEDER. For feeding dry chemicals continuously by weight, Omega Machine Co. introduces a new midget belt gravimetric feeder. Has 8-in. belt, a 1-ft. weigh span, capacity of 3 cu. ft./min., a feeding range of 100:1, variable speed transmission, hopper agitation, proportional feed rate and an alarm device.

2 • SELF-CLEANING FILTER. For processes where large amounts of solids are filtered, and where cleaning is difficult or inconvenient, a new self-cleaning filter has been introduced by the Sparkler Manufacturing Co. Cleaning is accomplished by a set of nylon brushes positioned between circular filter discs. To clean, water is flushed through the filter and the cage revolved so that the discs are swept by the nylon brushes. The filter is a series of circular discs mounted on a hollow center shaft and enclosed in a horizontal cylindrical pressure tank. Discs are perforated on sides and covered with a synthetic filter media sealed at the edge. Spacer rings hold the cloth in place and depths of the rings may be varied in accordance with the thickness of the cake desired.

3 • SPHERICONE. A radical design in sedimentation tanks for hot process water softeners, is a development of the Graver Water Conditioning Co. The new tank is a hemisphere on top of a cone, and was designed to provide a large settling area with low rising rates for the treated water. The inverted cone provides a uniformly increasing area from tip to circular top and the hemisphere provides the necessary structural strength. The raw water, steam and chemical inlets, spray heater, deaerator, and treated water collector are all mounted in the hemisphere, while the inverted cone contains the downtake, sludge bed.

4 • HCl PUMP. For handling hydrochloric acid, a new flex-i-liner pump

is in production by the Vanton Pump Corp. The pump is actuated by an eccentric rotor moving inside a flexible liner which in turn moves inside the pump block. This gives a squeezing action to the liquid, and a positive action. The liquid flows on the outside of the liner but on the inside of the pump block. The block can be of any material and for corrosive uses various plastics are recommended. The flex-i-liner is made of various elastomers both natural and synthetic. No stuffing boxes, self-priming, a maximum output of 5 gals./min. and pump sizes range from $\frac{1}{8}$ in. to 1 in. Operating speed is 750 rev./min. and the company has in development stage, larger pumps up to a capacity of 20 gals./min.

5 • STREAM-LINED GASKET. For reducing turbulence in pipe borne fluids at flanged and gasketed joints, the Flexitallic Gasket Co. offers a compression type with an inside and outside ring. Theory behind the new sealer is that by filling up all flange recesses, no turbulent eddies are set up at the joints. The inside ring converts one of a pair of smooth-faced flanges to a groove flange. It also provides the compression gage which prevents radial movement of the gasket. Made for all standard fittings and special designs—pressures from 150 to 2500

lbs. Inside ring of stainless steel, monel, nickel, etc.

6 • STEAM TRAP. A small, forged steel steam trap for high pressure superheated steam service from the V. D. Anderson Co. Suitable for pressures up to 500 lbs., 800°, and $\frac{1}{2}$ to $\frac{3}{4}$ -in. pipe connections. The forged steel head and body are safety measures to withstand shock and explosion.

7 • TEFLON GASKETS. Corrosion- and heat-resistant gaskets of Teflon were recently announced by the Chicago Gasket Co. Resistant to more than 200 acids, alkalis, etc., the new gaskets are fabricated in many different forms including both solid and Teflon-jacketed types. Available in hard, medium, and soft grades; produced in sizes from $\frac{1}{2}$ to 6 in. I.D.

8 • PULSATION DAMPENING. A pre-fabricated piping system which dampens pulses in gaseous systems, is a new service of The Fluor Corp., Ltd. Pulse-peak pressures, caused by reciprocating compressors, and source of trouble in metering accuracies, are eliminated by the new system. Volume bottles, laterals, and headers are utilized to provide the appropriate components of an acoustical filter. Fluor incorporates its dampening principle in initial compressor piping design. Can be used on any type of gaseous flow, air,

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steam, or hydrocarbons, etc., at operating pressures ranging from a vacuum to 5000 lbs./sq.in. at any temperature. Two main dampeners are utilized in the design. One is a package type for removing pulsation and vibration caused by reciprocating action of the compressors, and second, a higher frequency dampener which is recommended for installation in compressor piping systems where the pulse frequencies are in a range of the second harmonic.

9 • ANGLE DISINTEGRATOR. For the food and chemical processing industries Reitz Mfg. Co. offers an angle disintegrator which crushes, grinds, mixes, shreds, etc. Feature of the design is the demountability of working parts. Take-down assembly time is a matter of a few minutes. Sterilization of all working parts is possible. Dual discharge which separates the ground from the unground material. Belt drive. Rotor speeds from 7,200 to 16,000 rev./min., stainless steel, $\frac{1}{2}$ to 30 hp. Rotor diameters varying from 4 in. to 12 in.

10 • MELTING-POINT METER. The Clarkstan Corp. is in production with a heater for determining m.p.'s between 20° and 500° F. Used for waxes, resins, fats, tars, plastics, etc. Uses only a few milligrams. In

operation a small quantity of a material is placed on a chromium-plated platen which is electrically heated. Observation determines the melting point and the temperature read by a meter-thermocouple combination.

11 • RATIO TOTALIZER A totalizer which receives several control signals or pressures and produces a single output signal in any desired ratio to the incoming loads, is a development of the Hagan Corp. It is pneumatically operated and can control as many as three input loading pressures in a single unit. It is designed for use in computing circuits for such functions as addition, subtraction, multiplication, division of control signals, etc. Has been used in general process control, central station and industrial power plant regulation, aeronautical laboratories, etc. Accuracy to within 1% of full-scale reading.

12 • DUST COLLECTOR. A combination impingement, spray, and packed tower for the removal of dust from industrial gases, is being built by Prater Pulverizer Co. A steel shell collector, the dust-laden air enters the bottom of the shell tangentially and strikes the wetted walls of the shell. Air then forced upward through a series of throat plates, fitted with overhead jet spray nozzle.

Then through a bed of ceramic packing, also water-sprayed and finally through another bed of dry ceramic packing which acts as an entrainment separator.

14 • VISCOSITY MEASUREMENTS. For making precise measurements of viscosity in ultra-low ranges, Brookfield Engineering Laboratories are producing an adaptor for attachment to their viscometer. Essentially it is a cylindrical spindle mounted within a concentric tube. In measuring between 0 to 10 cps. reproducibility within .02 cps. possible. Essentially the adaptor provides for amplifying effects in viscosity measurement.

15 • VISCOUS-LIQUID PUMP. For handling highly viscous liquids, Schutte and Koerting Co. designed a new steam-jacketed, herringbone gear pump. The steam jacket completely covers all internal parts. Inlet, outlet and cover flanges cast integrally with pump base. The rotor and bearing assembly can be removed as a unit. Available in bronze, alloy, steel, etc.

16 • PLASTIC PIPE. Carlon Products Corp. has a plastic pipe furnished in threaded sections with molded plastic fittings. Lengths of 20 ft. can be threaded and cut, and elbows, tees, adaptors, plugs, etc., are available. Pipe sizes from $\frac{1}{2}$ in. up to 2 in., bursting pressures from 1000 down to 500 lbs./sq.in. for the same sizes.

17 • CARTRIDGE DEMINERALIZER. For use where corrosion, rust, etc., are problems, Penfield Mfg. Co. offers a new cartridge, ion-exchange unit which will furnish up to 10 gals./hr. of demineralized water. Has a flow meter for adjustment to proper rate. The unit is supplied with a permanent cartridge which is retained; the resin being discharged and replaced. Electric conductivity meter built into the unit gives indication of the quality of the water.

18 • CHLORINIZER. A high capacity chlorinizer for metering and controlling chlorine in control of slimes,

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chemical processing, water treatment, industrial waste treatment, etc., was recently exhibited by Builders-Providence, Inc. Has a visible gas flow indicator plus an automatic control to provide chlorine to three points of application.

CHEMICALS

25 • TERTIARY-OCTYLAMINE AND ALKYLAMINE 81. Rohm and Haas offers a 16-page technical description of tertiary alkylamine and octylamine 81, both primary aliphatics. The pamphlet follows the usual pattern of these books giving the properties and suggested uses, structure, physical constants, solubilities, surface tensions, etc. Possible applications as corrosion inhibitors, intermediates in the synthesis of insecticides, fungicides, wetting agents, anti-oxidants, oil additives, etc. Half the book is given over to typical reactions of the tertiary alkylamines.

26 • POLYVINYL METHYL ETHER. General Aniline & Film Corp. is in production with polyvinyl methyl ether available in 55 gal.-drums for use in pharmaceutical synthesis.

27 • GASES. A catalog and price list on gases have been published by The Matheson Co. Contains data on 74 rare and common gases, gives information, in addition to prices, on cylinders, facts about controls, regulators, cylinder markings, sizes, details of regulators, accessories, etc.

30 • 1,4-BUTANEDIOL. A bulletin giving properties, characteristics, and handling information for 1,4-butanediol which is available now in pilot plant quantities, from Antara Products, a division of General Aniline & Film Corp. Bibliography and chart showing products derived from the compound are included in the bulletin.

BULLETINS

35 • SLUDGE REACTOR. A description of the principle of contact

sludge reactor operation from the Cochrane Corp. Illustrated with pictures, line drawings, to show how the equipment operates. The bulletin also gives advantages, shows the various types of reactors with construction details, gives details of applications.

36 • CYCLON. A bulletin which explains Cyclon corrosion resistant materials of construction. Essentially a family of resins which Munray Products Inc. has formulated into organic coatings, chemical tubing and piping, process equipment linings, and custom formulations and fabrications. Suggested uses and materials are given.

37 • POROUS MEDIA. The Carborundum Co. in a large bulletin on porous media, covers the use of aluminum oxide in all its various applications. The book, well filled with photographs, charts, and tables of data, goes into the composition and structure, grades, physical properties, capillarity of the material, and then proceeds to itemize its uses in filtration and diffusion. An excellent exposition of the industrial applications.

38 • INHIBITING CAUSTIC CORROSION. For preventing the corrosive attack of caustic soda on glass surfaces, The Solvay Process Div. has

developed a new detergent Nytron. Results of the test are included in a 26-page report of a research project. Nytron was used in washing tests and the report summarizes and gives in detail the results obtained with 2% and 5% solutions of caustic soda.

39 • PROTECTIVE LININGS. The Glidden Co. tells the story of protective linings for steel drums and pails. The book catalogues various types of materials, and in each case it gives the type of protective lining suitable. Divisions are animal and vegetable oils, mineral oil production, strong solvents, food products, etc. Describes the coatings that are recommended.

40 • WEIGHERS. B. F. Gump Co., manufacturer of automatic net weighers for packaging, issues a pamphlet which covers feed regulators, gravity-feed weighers, power-feed weighers, installation accessories, etc. Covers the field of package weighing.

41 • DUST COLLECTOR. For the mining and metallurgical industries, American Wheelabrator & Equipment Corp. in a new bulletin describes the operation of cloth-tube collectors employed in recovering values from roaster gases, sintering machines, induction furnaces, etc.

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The bulletin lists some materials that have been successfully collected by the tube-type dust collector. Available with synthetic fiber cloth for corrosive gases at elevated temperatures.

42 • SARAN-LINED PIPE. A new catalog on Saran-lined steel pipe fittings and valves is available from the Saran Lined Pipe Co. The bulletin gives information about the advantages for uses in combating corrosion within the temperature limits of -40° to $+194^{\circ}$ F. Specifications are given for lined steel pipe, union fittings, flange fittings, etc. Valves, hose connections, sight flows, plus pictures of typical installations.

43 • HAMMER MILLS. Bauer Bros. Co. in a brief bulletin describes various models of hammer mills. Three series are described: one with a cyclone collector popular with feed, industrial, agricultural and forest product applications. The second, a low-speed mill which produces a minimum of dust and has reversible rotation and the third model designed for custom feed grinding machines. Pressure feeders and various types of magnetic separators are also briefly described.

44 • FILTERS. A description of the Titeflex line of filters. Filter member has permanent membranes and the completely assembled units contain precoat tanks, motors, pumps and filter chambers. Filter sizes run from 1 sq. ft. of filtering area up to 10 with concomitant capacities of 400 to 2240 gals./hr. Used in the electroplating, soap, wine, dry cleaning, drug, etc., industries.

45 • VIBRATING CONVEYOR. Stephens-Adamson Mfg. Co. describes a vibration-type conveyor which features a trough and frame

which vibrate in opposite directions. The vibration drive is through an eccentric shaft and connecting rod, the conveyor being mounted on springs. Conveying speeds up to 70 ft./min. for bulk and packaged material. The materials can be carried up inclines of 10° ; vibration has a $\frac{3}{8}$ -in. amplitude and a normal frequency of 800 strokes/min.

46 • STEAM GENERATORS. Use of a new principle in steam generation—dual circulation, developed by the Foster Wheeler Corp. is described. In principle the new steam generator utilizes two separate heat-absorbing sections each having its independent circulating system. Feed water is admitted to the primary section, and its blowdown is introduced to a secondary section as feedwater. Effect is to reduce silica carryover as well as steam dissolved hydroxides, chlorides, etc. Completely illustrated and replete with data and cutaway illustrations showing operations.

47 • SWEETLAND PRESSURE FILTER. A bulletin from Oliver United Filters, Inc., describing the Sweetland pressure filter brings information up to date. Applications are detailed as well as structural information on different sizes. These range from laboratory units with $1\frac{1}{2}$ to 5 sq. ft. of filtering area to units with 480-1000 sq. ft. Construction, operating cycle, as well as listing of the proper filters given.

48 • GRINDING AND MIXING EQUIPMENT. A complete line of grinding and mixing equipment of the U. S. Stoneware Co. Bulletin begins with a description of the new Borondum tubular grinding material, and then details of roller-type jar mills, tumble mixers, mills, fars, drum-rollers, tumblers, special mixing equipment,

etc. Well illustrated with detailed data frequently given.

49 • CENTRIFUGAL PUMP. DeLaval Steam Turbine Co. has a new GS pump with features of all parts contained in one replaceable rotor assembly. Three sizes from $2\frac{1}{2}$ in. to 4 in. for capacities to 400 gals./min. and heads of 230 ft.

50 • ANTI-CORROSION COATINGS. Minnesota Mining & Manufacturing Co. with information on adhesive sealers and coatings for protecting metals against acids, chemical fumes, solvents, etc. Contains the properties of more than 100 industrial adhesives and allied materials. Gives tables on coatings for protecting metal against deterioration, adhesives for bonding metals, sealers, etc. Physical characteristics of the adhesives, uses, etc.

51 • SAFETY HEADS. From Black, Sivalls & Bryson, Inc., a safety head price list explaining sizes, ratings, specifications and prices of safety heads and accessories. Also covered in the booklet are safety head flanges, rupture discs, vacuum supports, relief valves, etc.

52 • DUST COLLECTOR. A new 12-page catalog issued by the Ducon Co. describes industrial dust recovery with cyclone collectors. Operation of the cyclone is explained, and data are fractional efficiency curves, arrangement of multiple units. Other equipment made by the company in the same field is shown.

53 • STEAM HOOK-UPS. For the convenience of plant engineers in making the correct hook-up, their sixth, enlarged edition of the Sarco Steam Hook-Ups. It covers the correct steam trap selection and calculation for steam trap sizes and it has numerous pages of illustration showing how piping, strainers, traps, pumps, kettles, dryers, presses, etc., should be vented and drained. Discusses steam heating systems; gives calculations, discusses temperature regulators, valve sizing, etc. Tables of properties of saturated steam, flange standards, pipe threads, etc., round out the bulletin.

54 • VACUUM DATA. A publication, "Much Ado About Nothing," is being published by the Beach-Russ Co., which gives data, information and tips on vacuum processing through the use of vacuum pumps. Beach-Russ Co. manufactures rotary vacuum pumps and the bulletin which is published by it discusses vacuum technology.

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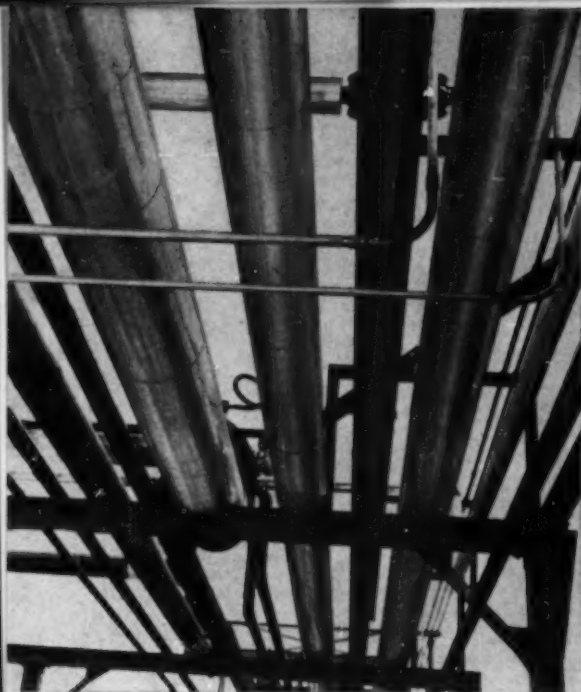
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Chemical Engineering Progress

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This close-up clearly shows the ribs or corrugations which add strength to Childers Jacketing and also make it form more easily around lines. Aluminum strapping and seals are a quick, inexpensive way to attach the jacketing. They can be used without any special tools and take no special training for the installers.

Does long lasting aluminum cost too much for covering your insulated lines? Not when you specify this new type weather-proof jacketing. It has been specially developed to cost little more than ordinary temporary jacketing materials.

New Low-Cost Jacketing Cuts Maintenance on Insulated Lines

AT LAST an aluminum jacket has been "engineered" especially to protect outdoor lines in chemical plants and refineries. It is the first "permanent-type" jacketing that offers both low initial cost and low application cost.

After two years of tests and major installations at 431 outdoor locations in chemical plants, refineries, and power plants, the Childers Manufacturing Co. of Houston has rung up an impressive cost-cutting record with their Childers Aluminum Jacketing.

Secret behind this low cost is the thin aluminum sheet—.006" thick—which is the weather-protecting sheath of the jacketing. It is ribbed (as pictures show) for extra strength and to facilitate forming around the line. This aluminum has

proved thick enough to resist weathering and usual abrasion, but costs much less than heavier aluminum sheets.

Easy application of this Childers Jacketing cuts labor bills drastically. It comes from the factory in easy-to-handle rolls that are four feet wide and either 100 feet or 200 feet long. It is light and flexible to handle as the men put it on the line. No roll forming required as with heavy, un-crimped material. It can be cut off and attached just the same as the old-fashioned tar-paper that was sometimes used for jacketing.

You can attach Childers Jacketing around your insulated lines by one of several easy methods. The use of aluminum strapping and seals is probably the best. Sheet metal screws or Minnesota Mining's No. 471 tape can also be used.

The Childers jacketing can be removed and re-used.

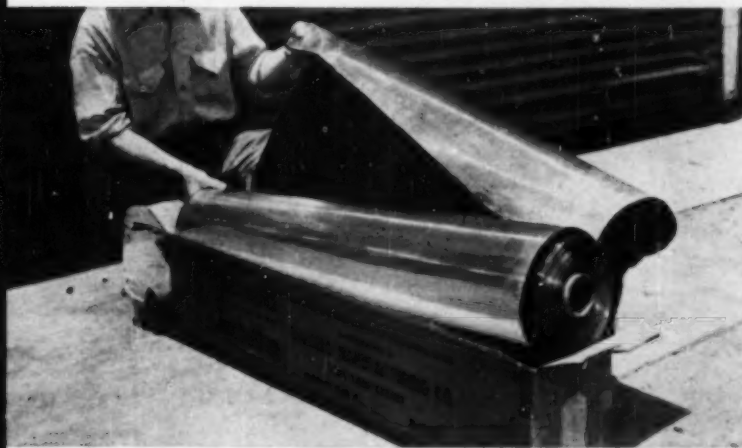
A moisture barrier is attached to the back of the jacketing to give positive protection for the aluminum when it is used with alkaline insulating materials.

No painting, no rust-proofing, little maintenance—that's the advantage of aluminum!

Hundreds of design engineers and maintenance engineers have agreed they save money for their firms and trouble for themselves by changing their jacketing specs to Childers 100%. That should make it worth your testing too—and a test roll costs only \$36.00 for 400 sq. ft. (with moisture barrier attached—price for jacketing without moisture barrier much less). That's a good investment in any plant. (Advt.)

★ ★ ★

FOR FULL INFORMATION write Childers Manufacturing Co., Dept. CP-1, 625 Yale St., Houston 7, Tex., for complete literature. Childers has engineering representatives in principal cities to work with you on specific problems.



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COASTAL GEORGIA CHEMICAL ENGINEERS CLUB

The third meeting of this club was held Sept. 15 in Brunswick. A total of 55 persons attended from Savannah, Brunswick, Waycross and Helena, Georgia, and Jacksonville, Florida. Included in these were 11 members of the Savannah Chapter of the American Society of Mechanical Engineers.

Speaker for the occasion was J. E. MacConville, Atlanta regional manager, Brown instruments division, Minneapolis Honeywell Regulator Co. Mr. MacConville talked on "Graphic Panel Instrumentation."

The following officers were elected to serve until Jan. 1, 1952.

Chairman—H. J. Hainer, Hercules Powder Co., Brunswick
Vice-Chairman—R. R. Chase, Union Bag & Paper Corp., Savannah
Secretary—E. O. Barnes, Union Bag & Paper Corp., Savannah
Treasurer—J. B. Loucks, Union Bag & Paper Corp., Savannah
Executive Committee—(in addition to officers)
J. R. Lientz, Union Bag & Paper Corp., Savannah
A. R. Bookout, Hercules Powder Co., Brunswick
B. P. Allen, Southern Paperboard Corp., Port Wentworth

Reported by E. O. Barnes

PHILADELPHIA-WILMINGTON

The first meeting of the 1950-51 season was held at Clubhouse Hotel, Chester, Pa., Oct. 10, with 90 at dinner and 150 at the meeting. They heard H. M. Schmitt, chemical industry manager, of Brown instruments division, Minneapolis Honeywell Regulator Co., on the subject "New Types of Instrumentation for Chemical Processing."

Mr. Schmitt gave an illustrated review of many developments in chemical process control by instruments of high sensitivity in many fields.

A plant trip was made to the Eagle Point works of The Texas Co. at Westville, N. J., Oct. 21.

Reported by R. B. Chillas, Jr.

AKRON

This section met Oct. 12, 1950, at the University Club. About 60 members and guests attended the meeting.

Dr. Warren L. McCabe, President of A.I.Ch.E. and vice-president of Flintkote Co., spoke to the group on the functions and activities of the Institute. Dr. McCabe also showed many slides, one in particular depicting the growth of A.I.Ch.E.

Reported by J. W. Kosko

(Continued on page 40)

MARGINAL NOTES

(Continued from page 22)

Chemical engineers are accustomed to a more extensive treatment of generalized properties with their accompanying charts. In this first edition the generalized compressibility charts are inconvenient for actual calculations in view of oddly chosen parameters and somewhat poor reproduction. Likewise, the space afforded applications of high pressure behavior is mainly by reference. There is also lacking any treatment of excess thermodynamic properties incorporating the work of van Laar, Margules, and Scatchard-Hamors. However, these are points of distinction between two fields of interest and do not detract in any way from Professor Rossini's contribution.

The author has wisely included a most generous system of references for collateral reading.

The book has been carefully proof-read, only two obvious typographical errors—Equation (17) on page 266 and the misplacement of the exponent in Equation (19), page 107.

Many Opinions on Combustion Phenomena

Third Symposium on Combustion and Flame and Explosion Phenomena. Published under auspices of the standing committee on combustion symposia by Williams and Wilkins Co., Baltimore, Md. (1949) 748 pp. \$13.50.

Reviewed by Walter E. Lobo, Director, Chemical Engineering Department and J. B. Dwyer, Chemical Engineer, The M. W. Kellogg Co., New York, N. Y.

THIS volume presents 100 papers covering the recent research, both experimental and theoretical, in the fields of combustion and explosion. Hence it is neither a textbook nor handbook, but a reference source. Each author presents in his own way results of his work in one of the many phases of the combustion field.

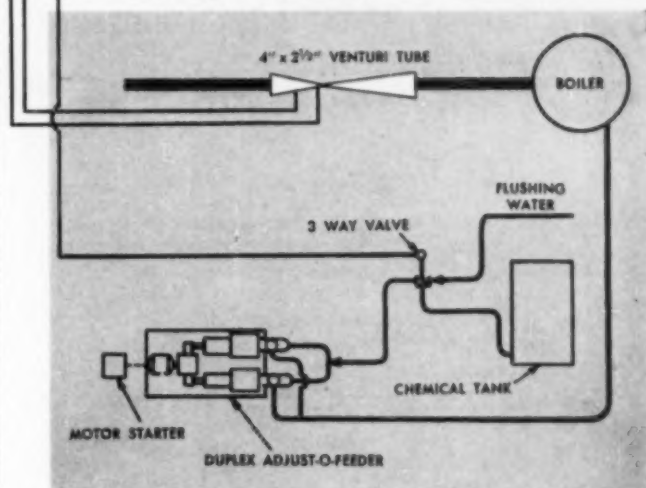
Dr. Bernard Lewis, in his introduction to the symposium, points out that while only three major fields of investigation remain (chemistry and kinetics, ignition and rate of flame propagation, and thermodynamics) these have now been subdivided further and further with advancements in knowledge and techniques.

Most of the papers concerning kinetics and mechanism of combustion reactions are devoted to consideration of hydrocarbons, but oxidation of ethers, metal alkyls, carbon, carbon monoxide, sulfur dioxide and hydrogen are among the other subjects of papers. Several



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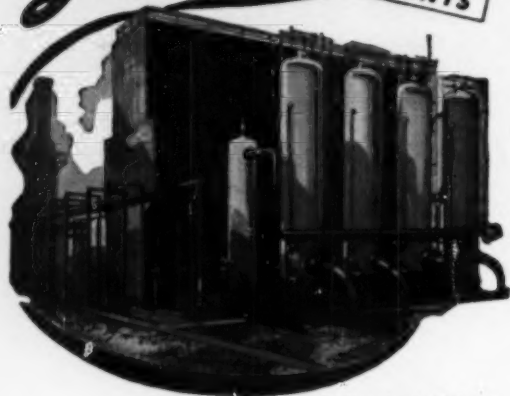
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papers are devoted to free radicals, their determination, etc. Techniques and results of investigations employing spectroscopic and radiometric methods are presented.

Ignition of combustible mixtures by electric sparks and by hot bodies is treated in a number of papers, and together with those on flame propagation, stabilization, and quenching they comprise more than one third of the text. A few papers are devoted to diffusion flames, both laminar and turbulent.

Subdivisions covering titles of the papers in the remaining one third of the book are "Burning and Detonation of Explosives," "Thermodynamics of Flame Gases and Thermochemistry," "Experimental Techniques," "Combustion in Engines and Rockets," and "Gas Burners and Furnaces."

Although there is considerable value to the preservation of the points of view and lines of attack of the various authors when a symposium of this type is published, there are certain drawbacks (insignificant to the expert but of importance to one not too familiar with the field) in such a presentation. Lack of uniformity of nomenclature is evident. The extent of condensation of the papers, so necessary to present them written in a few pages or spoken in a short period, has forced the authors to present only the essence of their work.

Had some introductory lectures, covering basic theory, background, and definition of terms been presented at the symposium, they would have added immeasurably to the understanding and appreciation of this fine work.

The Journal of the Imperial College Chemical Engineering Society, Vol. 4 (1948) 207 pp. + xix.

THIS volume includes the following articles:

The Use of Graphical Methods in Chemical Engineering Calculations
The Calculation of Particle Terminal Velocities
The Fluid Catalyst Technique in Modern Petroleum Refining
Liquid-Liquid Extraction; Part I; Centrifugal Methods of Extraction
Valves for Process Plant
The Displacement of Liquids from Porous Solids
Technology and Chemical Engineering Design
The Unit Operation of Mixing; Part I; Methods of Assessing the Performance of Agitators
The Unit Operation of Mixing; Part II; A Study of the Rate of Mixing of Solid Particles
Studies for the Design of Gas Lift Pumps; Part I; The Formation and Characteristics of Gas Bubbles
The Design of Heat Exchangers for Low-Temperature Plant
Research in the Department of Chemical Engineering and Applied Physical Chemistry

Chemical Engineering Reports. Kenneth A. Kobe. Third Edition. Hemphill's Bookstore, Austin, Tex. (1950) 67 pp. + Appendix.

DR. KENNETH A. KOBE, professor of chemical engineering at the University of Texas, has prepared a report to illustrate to chemical engineering students, the format for a chemical engineering report. He has written it in the form of a report on the literature of chemistry and chemical engineering.

The book is divided into eight parts: The Chemical Literature, where the various magazines and essential books for chemical engineering are listed in some detail; Engineering Reports which covers essential parts, types and form of presentation; The Formal Engineering Report; The Laboratory Report; Mechanical Preparation; Literature Citations; Literature Cited; and General Bibliography.

Books Received

A German-English Dictionary for Chemists. Austin M. Patterson. Third Edition. John Wiley & Sons, Inc., New York. (1950) 541 pp. \$5.00.

Antihistamines. Industry and Product Survey. Nathan Wisniewsky. Chemonomics, Inc., New York, N. Y. (1950) 157 pp. \$5.00.

Electrical Engineers' Handbook—Electric Communication and Electronics. 4th Edition. Editors: Harold Pender and Knox McIlwain. John Wiley & Sons, Inc., New York, N. Y. (1950) \$8.50.

Modern Chemical Processes. I & E C staff reports. Reinhold Publishing Corp., New York, N. Y. (1950) 222 pp. \$4.00.

Organic Syntheses. Vol. 30. Editor in Chief: Arthur C. Cope. John Wiley & Sons, Inc., New York, N. Y. (1950) 115 pp. \$2.50.

Organophosphorus Compounds. Genady M. Kosolapoff. John Wiley & Sons, Inc., New York, N. Y. (1950) 376 pp. \$7.50.

Primary Batteries. George Wood Vinal. John Wiley & Sons, Inc., New York. (1950) 336 pp. \$5.00.

Response of Physical Systems. John Dezendorf Trimmer. John Wiley & Sons, Inc., New York, N. Y. 268 pp. \$5.00.

The Economics of the Printing Ink Industry. Robert S. Aries and Anthony Nickolaus. Chemonomics, Inc., New York, N. Y. (1950) 108 pp. \$5.00.

The Journal of the Institute of Metals. Vol. LXXV. Published by The Institute of Metals, London. Editor: Lieut.-Colonel S. C. Guilan. (1949) 1230 pp.

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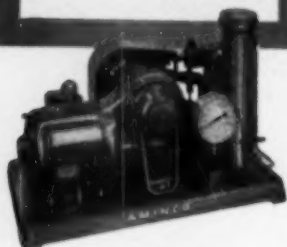
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COLUMBUS STORY

(Continued from page 19)

Diversity is a keynote to research today. As countless new products have poured forth from the nation's laboratories, new and highly diversified industries all over America have sprung up to manufacture and distribute them.

The Columbus area is no exception. Engineers attending the Columbus meeting in December will have an opportunity to visit industries whose classifications read like a series of titles from an industrial chemistry book. Some of the fields covered: water and sewage disposal, fertilizers, lime, sulfuric acid, oils, paint and pigments, plastics, synthetic organic chemicals, food, petroleum processing, electroplating, glass, paper manufacture, and process equipment manufacture.

According to the Ohio Development and Publicity Commission, more than a hundred-million dollars have been spent for chemical plants in Ohio since the end of World War II.

A total of 29 plants comprising 18 trips is scheduled for the convention. Exact details of these trips can be obtained from the official program distributed by the A.I.Ch.E. Office of the Executive Secretary.

LOCAL SECTION NEWS

(Continued from page 36)

KNOXVILLE-OAK RIDGE

A series of chemical engineering seminars is being arranged through the November period to continue the program which instituted a series of such seminars starting in June, 1950. Each summer a group of science and engineering professors from southern universities and colleges go to Oak Ridge as research participants under the auspices of the Oak Ridge Institute of Nuclear Studies. Several of the professors of chemical engineering in this group were invited to participate on the local section seminar program.

Four chemical engineering research participants at the Oak Ridge National Laboratory during the past summer presented brief talks and led discussions on four different evenings on the following:

1. "Some Diffusional Aspects of Solvent Extraction" by Dr. F. P. Pike, North Carolina State.
2. "Filtration" by J. I. Stevens, Vanderbilt University.
3. "Evaporation" by Dr. F. A. Anderson, University of Mississippi.
4. "Effect of Temperature and Pressure on the Viscosity and Thermal Conductivity of Gases" by Dr. J. M. Lenoir, University of Arkansas.

Reported by A. C. Jealous

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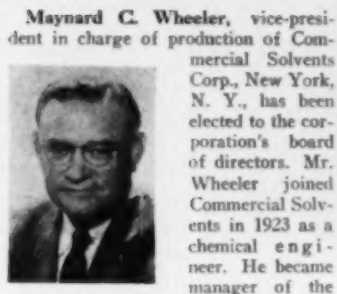
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PEOPLE



Maynard C. Wheeler, vice-president in charge of production of Commercial Solvents Corp., New York, N. Y., has been elected to the corporation's board of directors. Mr. Wheeler joined Commercial Solvents in 1923 as a chemical engineer. He became manager of the

Terre Haute plant in 1929, and later, production manager for all Commercial's plants. In 1945 he was made vice-president in charge of production. Mr. Wheeler, a director of Thermoatomic Carbon Co., Sterlington, La., is an alumni counselor of the Purdue Research Foundation.

STUTZMAN, WILLIAMS IN NORTHWESTERN CHANGES

Dr. Leroy F. Stutzman, associate professor since 1948, has been appointed chairman of the department of chemical engineering at Northwestern University's Technological Institute, Evanston.

Dr. Stutzman joined Northwestern in 1943 as assistant professor of chemical engineering. Prior to going there, he was director of rubber research for the Pittsburgh Coke and Iron Co. His earlier experience includes assignments with the Mellon Institute, Kansas State College and Purdue University. Dr. Stutzman received his Ph.D. from the University of Pittsburgh in 1946.

In his new position Dr. Stutzman succeeds Virgil C. Williams who resigned to accept the appointment of associate director of General Aniline & Film Corp.'s central research laboratory at Easton, Pa. Mr. Williams will also be in charge of chemical engineering research and development.

Raymond W. McNamee was recently named superintendent of the research and development department of the Carbide and Carbon chemicals division, Union Carbide and Carbon Corp. at South Charleston, W. Va. He had been serving at acting superintendent. Dr. McNamee has been connected with the department since 1933.

W. E. Keppler, formerly with Heyden Chemical Corp., is now with the engineering division of Merck and Co.



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Knight also offers a light weight laminated plastic under the trade name of Permanite. It is corrosion resistant throughout and is used in the fabrication of complete functional units, such as fume washers, HCl absorbers, etc. Permanite is also available in light weight pipe and duct work.

When making inquiry please specify the type of equipment in which you are interested as well as service conditions involved. Literature will be sent on request.

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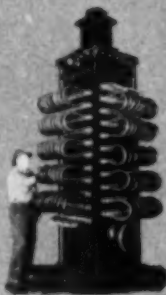
Acid and Alkali-proof Chemical Equipment



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HCl Absorbers, Towers



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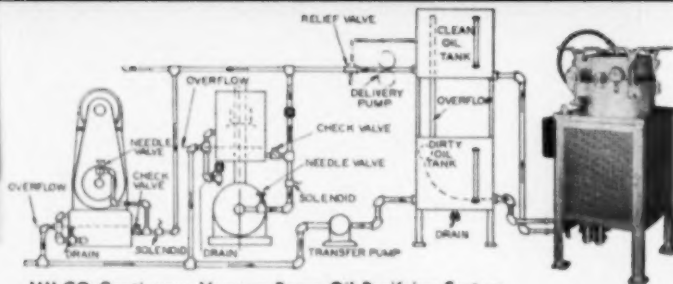


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Oil purified at a fraction of a cent per gallon.

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John R. Riley, Jr., formerly general sales manager of Spencer Chemical



Co., Kansas City, Mo., has been elected vice-president in charge of sales. Mr. Riley is a graduate chemical engineer. Joining the firm in 1942, he served first in the operating division and then

headed up postwar utilization studies. When the Spencer company went into commercial operation, Mr. Riley took charge of the sales division. Prior to 1942, he was employed by the Shell Petroleum Corp. and Dearborn Chemical Co.

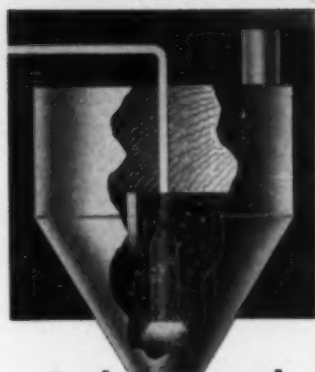
HUTCHISON, BRANCH MGR. SALES, MONSANTO

J. C. Hutchison, formerly assistant branch manager of Monsanto Chemical Co.'s Detroit district (sales), located in Cincinnati, has been appointed branch manager effective Jan. 1, 1951. This is in line with the company's recent announcement that the Detroit, Cleveland and Cincinnati sales territories of the organic chemicals division will become separate districts. Mr. Hutchison will manage the new district at Cincinnati.

Mr. Hutchison is a native of Brownsville, Tenn. He received a B.S. degree in chemical engineering from the University of Tennessee in 1935 and joined Monsanto in 1941. He became a member of the sales engineering department in 1942 and was transferred to Cincinnati in March, 1949, as an assistant branch manager of the Detroit district.

Richard S. Egly has been appointed director of chemical research in the research & development department of Commercial Solvents Corp., at Terre Haute, Ind. Formerly in charge of chemical engineering research, Dr. Egly has supervised work on many of the corporation's products, including nitroparaffins, methylamines, and benzene hexachloride. Dr. Egly received a B.S. degree in chemical engineering from Purdue in 1936, and a Ph.D. in chemical engineering and organic chemistry from the University of Illinois in 1940. He joined Commercial Solvents after receiving his doctorate.





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Souren Z. Avedikian, president and technical director of Tidings Corp. of America, Larchmont and Mamaroneck, N. Y., was recently awarded a "Commendation for Meritorious Civilian Service for Outstanding Performance of Duty" by the U. S. Army. Dr. Avedikian served as senior chemical engineer from 1940 to 1947 at the New York Chemical Procurement District. For four years prior to 1947 he was assigned at Edgewood Arsenal, Maryland.



William T. Cravens, assistant general superintendent, Cities Service Oil Co., Bartlesville, Okla., has been named general superintendent. Early in his career he was associated with the Arkansas Natural Gas Co. and the Arkansas Fuel Oil Co., both subsidiaries of Cities Service Co. at Shreveport and following a short service period with the Girdler Corp. he became associated with the Cities Service Refining Corp. at Lake Charles, La. From 1942-46 he was chief process engineer and supervisor of processing, engineering department, Cities Service Refining Corp. at Lake Charles, an assignment he left in December, 1946, when he was appointed chief process engineer, manufacturing and research department of Petroleum Advisers, Inc., a New York subsidiary of Cities Service Co. Mr. Cravens is a graduate of the University of Arkansas with a B.S. degree in chemical engineering.

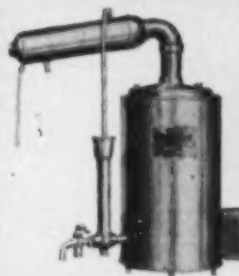
Melbourne L. Jackson, assistant professor in the chemical engineering department of University of Colorado, Boulder, Col., has been granted a year's leave to work in the U. S. Naval Ordnance Test Station at China Lake, Calif. Dr. Jackson's leave inaugurates the



chemical engineering department's new policy of asking faculty members to accept periodically a year's employment in nonacademic fields. Dr. Jackson has been at the university since 1948. He has a B.S. in chemical engineering from Montana State College and a Ph.D. from the University of Minnesota. He taught at both universities before going to Colorado. He has done research work in the fields of fluid dynamics and the use of radioactive tracers.

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*Article in C. & E. News Vol. 27 - 279

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R. L. Demmerle was recently appointed executive editor of Chemical Industries, acquired by the McGraw-Hill Publishing Co. Mr. Demmerle will continue in his post as associate editor of Chemical Engineering. For the five years prior to his joining the McGraw-Hill organization, he was New York editor of Chemical and Engineering News and Industrial and Engineering Chemistry.



A. P. COLBURN, PROVOST AT DELAWARE UNIV.

Allan P. Colburn has been appointed to the newly created administrative post of provost at the University of Delaware, Newark, Del. Since April 1 he has been the university's acting president.

The new provost joined the University of Delaware faculty after nine years' service with the Du Pont Co. as a research chemical engineer. He became consultant for that company early in World War II. Since his association with the University of Delaware in 1938 he has been an associate professor of chemical engineering for three years, and has held the rank of professor since 1941. For nine years he was chairman of the department, leaving this post when he was named assistant to the president and adviser on research in 1947. In 1948 he received the Professional Progress Award in Chemical Engineering—its first recipient. Twelve years earlier the A.I.Ch.E. presented him with the Walker Award for publications in the field of chemical engineering. He has served as a director and chairman of the Awards Committee and as chairman of the Publications Committee of A.I.Ch.E.

David S. Weddell, director of development for Monsanto Chemical Co.'s Western division at Seattle, Wash., will rejoin the company's general development department in St. Louis. Dr. Weddell, who assumed his Western division responsibilities in March, 1949, joined the company in 1941 after receiving a Sc.D. degree in chemical engineering from M.I.T. After serving as a chemical engineer in the phosphate division's research department at Anniston, Ala., he was transferred to the central research department at Dayton, Ohio, then returned to Anniston as a group leader in research and joining the general development department in March, 1947.

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Milton W. Gregory has joined Glass Fibers, Inc., Waterville, Me., as sales manager of the company's bonded mat division. A chemical engineering graduate of Purdue University in 1940 Mr. Gregory was sales engineer for five years for the De Vilbiss Co.



He was also employed by the Du Pont Co. as a process engineer supervisor and by the Firestone Tire & Rubber Co., as a chemical process engineer.

Edward A. Mason since this past summer has been serving as professor of chemical engineering at M.I.T. where he was awarded an Sc.D. degree in chemical engineering in June. His present duties are those of the director of the Bangor Station, school of chemical engineering practice. This is located at the Eastern Corp., Bangor, Me.

W. E. Patterson, manager of engineering and development, Merck & Co., Limited, Montreal (Que.), Canada, was appointed director of publications of The Chemical Institute of Canada at a recent meeting of the board of directors of the Institute. Mr. Patterson's duties will include supervision of the Institute's monthly journal, "Chemistry in Canada," and any of the other activities of the Institute of a public relations nature.

John Bogen, formerly with the production department of the Pennsylvania Salt Manufacturing Co., in Philadelphia, Pa., and with the staff of the Calvert City (Ky.) plant, has been transferred to Wyandotte.

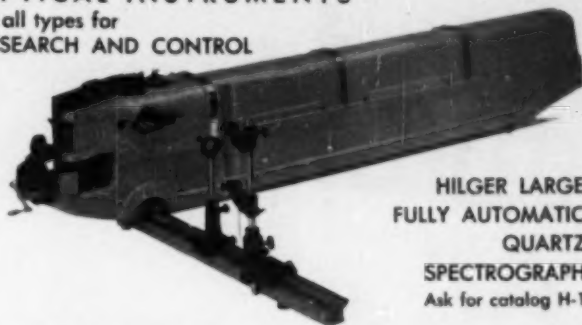
R. K. MUELLER ADVANCED BY MONSANTO

Robert K. Mueller has been appointed assistant general manager of the plastics division of Monsanto Chemical Co., Springfield, Mass., effective Oct. 15.

Mr. Mueller, who has been serving as division production manager, has been with the company since 1935. A graduate of Washington University, he holds an M.S. degree from the University of Michigan. He served in the production department of Shawinigan Resins Corp., and during World War II was general superintendent and later plant manager of the Longhorn (Tex.) Ordnance Works. A native of St. Louis, he joined the plastics division in 1946 and became successively assistant production manager and production manager.

(More about People on page 47)

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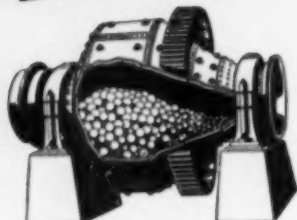
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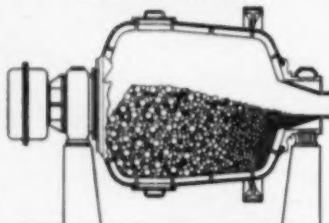
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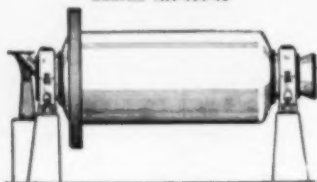
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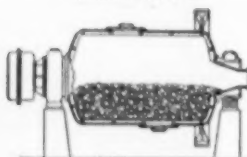
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WANTED—Mechanical Engineer with 8-10 years' experience in the design of equipment for chemical plants. Location Western New York. Box 4-11.

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Chemical Engineer—B.S. 1944, M.I.T. Age 28. Over six years' experience petroleum refining, including foreign service; manufacturing, design, and development problems. Desire position involving plant operations or liaison between plant and development/design. Box 1-11.

Development or Technical Service—Seven years varied research and development on corrosion, electrochemistry, and non-ferrous metallurgy of the newer unfamiliar metals at leading non-profit research organization. B.S.Ch.E. Married, 30, no physical defects. Box 5-11.

Research and Development Chemical Engineer—Ph.D. Age 28. Five years' varied experience in petroleum, tonnage oxygen, rocket, teaching fields. Patents, publications, honor societies. Desire challenging position with future. Box 6-11.

Technical Sales—Fifteen years' responsible experience production, engineering, sales. Last five years sales. Desire progressive sales position. Licensed engineer. Box 7-11.

Chemical Engineer—M.S.Ch.E. Eight years' varied experience major oil refinery. Technical service, process design, light ends process foreman. Excellent record cost reduction and efficiency improvement. Desire Midwest or Northeast location with medium or small refinery. Box 8-11.

B.S.Ch.E.—1948, 1½ years' experience in plant development: economics, design, purchasing. Seek position in research or development. Will relocate. Available mid-November. Box 9-11.

Chemical Engineer—B.S. and Bachelor of Laws—Qualified in petroleum refining and natural gasoline as follows: engineering, operations, management, and industrial relations. Seeking activity and unlimited outlet for ability. Full details furnished on request. Box 10-11.

Chemical Engineer—B.S.Ch.E. Carnegie Institute of Technology, 1949, married, 23, veteran. Presently employed. One year experience stream pollution abatement. Also trained and experienced in electronics. Engineering experience and advancement more important than initial salary. Box 11-11.

SITUATIONS WANTED

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Research Supervisor—Ph.D. Nineteen years' experience in petroleum derivative research and coordination of product development with manufacturing and sales. Presently director of small petrochemical research and petroleum product development laboratory. Want relocation in research, supervision or administration. Box 12-11.

Chemical Engineer—Five years maintenance supervision, equipment selection, design and estimating. Four years Naval engineering officer. Desire responsible position in maintenance, project engineering, or production. Will relocate. Box 13-11.

Sales Engineer or Manufacturers' Agent—M.S.E. Univ. of Mich. Experienced in sales to industry and dealers. Married, veteran. Presently employed. South-eastern Michigan. Prefer single line, but will consider combining two related lines. Box 14-11.

PEOPLE

(Continued from page 45)

Paul E. Burchfield, assistant superintendent in the development division of the Wyandotte Chemical Corp., Wyandotte, Mich., has been appointed director of technical service of the company's Michigan alkali division. Early in his career he was associated with the Harshaw Chemical Co., as a research chemist. Following a service period with the United Gas Improvement Co. he went to Wyandotte Chemical in 1943 and spent three years as assistant

Chemical Engineer—B.S. Age 27, veteran. One year experience in research. Three years industrial experience in petroleum refining—blending, treating, distillation and other related fluids. Presently employed. Available after January, 1951. Box 15-11.

Process Engineer—B.S.Ch.E. 1941. 30, veteran, married. 3½ years development and design heavy chemicals, including 1 year production supervision. 4½ years food industry, including 3 years group leader modernization program. 1 year varied consulting work. Box 16-11.

Chemical Engineer—B.S., 1941. Diversified process control, development, production, supervision, pilot plant. Last three years dyestuffs, intermediates. Presently located in Pennsylvania. Married; references. Available immediately. Box 18-11.

Chemical Engineer—M.S., Michigan, 1948. Age 29. Three years development and process engineering in plastics and heavy chemicals. Wish to remain in East. I can be of service. Box 19-11.

supervisor in the chemical engineering department, research division. He has been serving as assistant director of the technical service department since 1946. He has authored articles on such chemicals as styrene, indene and chlorobenzenes.

L. E. Gressingh, formerly chemical engineer with the Dasher Rubber & Chemical Co., Fairport Harbor, Ohio, is now associated with the H. R. Bowers Co., Cleveland, Ohio. He is serving as sales engineer.

Barrett S. Duff, who has been a graduate student at M.I.T., Cambridge, Mass., is now associated with the Pan American Refining Corp., Texas City, Tex. His title is chemical engineer.

Alan S. Michaels, formerly assistant professor of chemical engineering at M.I.T., has recently accepted a position as assistant technical director of the Seco Venture, Cambridge, Mass.

George A. Cristy is now senior design engineer at the Oak Ridge (Tenn.) National Laboratory. He was formerly production superintendent of the McCarthy Chemical Co., at Winnie (Tex.) chemical plant.

John M. Deal, formerly chemical engineer with the Union Oil Co. of California, Los Angeles, Calif., is now resident engineer with the same company located at Seattle, Wash.

Barnett F. Dodge, professor of chemical engineering, Yale University, New Haven, Conn., has been granted an award under the Fulbright Bill as a special lecturer at the University of Toulouse, Toulouse, France, for the second semester. This school has just established there an Institute of Chemical Engineering. Only a small number of students who are roughly equivalent to men with a B.S. degree in this country will be admitted. Professor Dodge will sail on the Liberté of the French Line Jan. 20, 1951.

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BEZANSON HEADS CHEMSTRAND CORP.

Osborne Bezanson, vice-president and member of the executive committee of Monsanto Chemical Co., became president and a member of the board of directors of the Chemstrand Corp. effective Nov. 15 (see p. 16). He will resign from Monsanto.

Mr. Bezanson has spent his business life with Monsanto and its predecessor company, the Merrimac Chemical Co., which Monsanto acquired in 1929. Practically his entire career has been in production and general management. Under his direction, Monsanto built and operated for the United States Government the Long Horn Ordnance works at Karnack, Tex., and the Texas City styrene plant during the war.

In 1942, he became a vice-president and member of the board of directors of Monsanto. The following year he was appointed general manager of the company's organic chemicals division, serving in that capacity until October, 1948, when he was elected to the five-man executive committee.

COM. SOLVENTS PROMOTES BULLS, DORSEY

Promotions in the staff of the research and development department of Commercial Solvents Corp., New York, N. Y., include the following:

James W. Bulls, formerly chemical engineer in charge of the research group at the Sterlington (La.) plant has been placed in charge of chemical engineering research at Terre Haute, Ind.

John J. Dorsey, who was previously in the chemical engineering group at Terre Haute, will replace Mr. Bulls at Sterlington.

JUDEFIND, TONGUE IN NEW ROLES WITH DAVISON

New assignments for members of the operations, and research and development divisions of the Davison Chemical Corp., Baltimore, Md., include the following:

W. L. Judefind was appointed assistant to Dr. G. M. Hebbard, vice-president of operations. Dr. Judefind will be stationed in the city office of the corporation.

T. O. Tongue was appointed manager of process and quality control at the Curtis Bay plant.

J. Henry Rushton, head of the department of chemical engineering, Illinois Institute of Technology, has been invited to give this year's lecture series for the Humble Oil and Refining Co., Baytown, Tex. The lectures will cover a two-week period and will be given to the members of the research and development division of the Humble organization.



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Announcing the Arrival of "miniPump"

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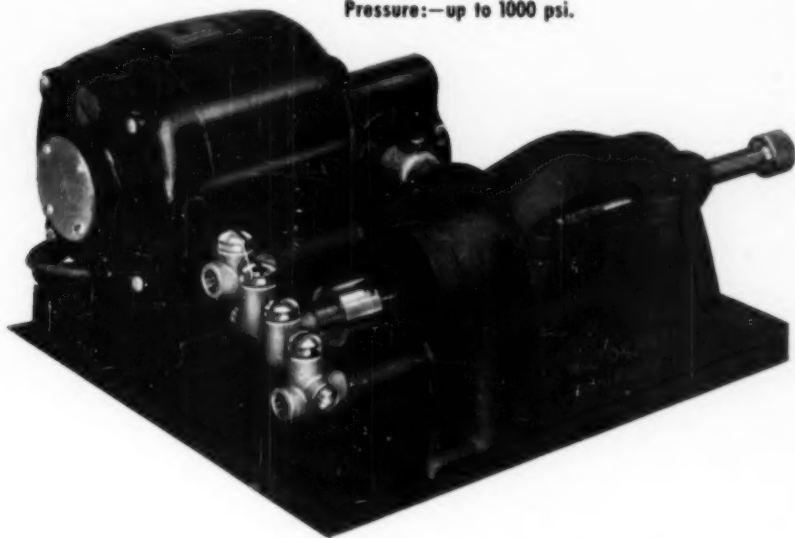
Mother: —the necessity for precisely metering and pumping liquids in minute quantities.

Father: —the one and only Milton Roy double ball-check design.

Weight: —42 lbs.

Capacity: —3–800 ml/hr

Pressure: —up to 1000 psi.



"miniPump" handles clear liquids in minute quantities at pressures up to 1000 psi. It is a true Controlled Volume Pump, incorporating the exclusive Milton Roy double ball-check liquid end in minified form with a fractional hp motor driver. As with all Milton Roy Pumps "mini" is a positive displacement, precise metering unit.

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The V/A Cell presents this fresh approach to fluid metering. It embodies important advantages of the area meter and the orifice meter, and eliminates numerous disadvantages of each. It provides, for instance, a **wide flow range** and a **linear scale** in the same instrument. It is a differential pressure instrument without need for mercury, bellows, seals, exterior purges, condensate chambers, or elaborate manifold systems. By making use of standard and well-established orifice data, the V/A Cell is simple to specify and to use.

V/A means "Variable-Area Metering Principle"

SPECIFICATIONS

Body Material—316 Stainless Steel
Other Parts in Contact with Fluid—316 Stainless Steel
Total Range—50" to 1400" H₂O Differential
Transmitted Pressure—3-15 PSI, Linear with Flow.

(Note: This unit is also available with Directly-Coupled Electronic Impedance Bridge Transmitter.)

Air Consumption—0.3 CFM

Working Pressure Ratings:

Series 60 Model
1040 PSI @ 350° F
1200 PSI @ 100° F

Maximum Operating Temperature—350° F

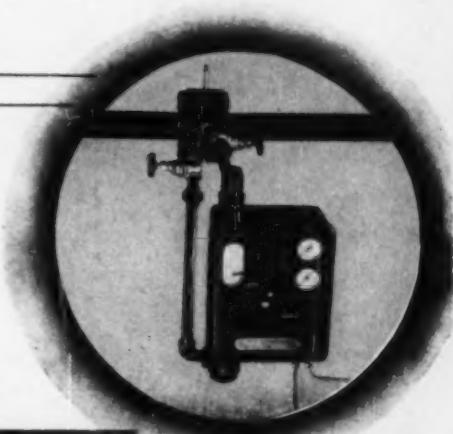
Air Supply—17-22 PSIG

Case—Moisture and Fume-Proof—Black Crackle Finish

Mounting—At Orifice Flange or up to 60 ft. Distant.

Primary Orifice Installation—In accordance with ASME or AGA Standards

- mercuryless
- flow indication at the orifice
- 50" to 1400" range
- linear response with flow
- inherent overrange protection
- no fluid level adjustments to make
- all 316 stainless in contact with fluid
- automatic continuous purge
- no elaborate manifold required
- no mechanical linkage to float
- no seal pots needed for corrosive services
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